Fatigue life enhancement of cast Mg alloy by surface modification in cold spray process

S.K. Shaha, S.B. Dayani, H. Jahed*

Department of Mechanical & Mechatronics Engineering, University of Waterloo, 200 University Ave W, Waterloo, ON N2L 3G1, CANADA

Abstract. To improve the fatigue life of Mg alloy, high strength AA7075 spherical powder was deposited on AZ31B samples in nitrogen carrier gas environment at 400°C temperature in cold spray process followed by electrostatic painting with zinc phosphate. The fully reverse four-point rotating-bending fatigue tests were conducted on the coated and uncoated samples in different environmental conditions. It is seen that the cold sprayed AA7075 improved the yield strength of the AZ31B cast alloy. Similarly, a significant fatigue enhancement was observed in the coated samples, reaching a fatigue strength of 90 MPa compared to as-cast fatigue strength of 70 MPa at 10^7 cycles when tested in air, and fatigue strength of 80 MPa under a 3.5%NaCl testing environment. The SEM analysis at the interface of the tensile tested sample exhibited the interfacial fracture followed by delamination of the coating. Similarly, delamination of coating was merely detected in the fatigue fracture sample tested in the air, while the sample tested in the corrosive environment showed pits which allowed the solution to penetrate in the AZ31B substrate results delamination and premature failure. However, the presence of e-paint forms a passive layer which is hindering the pit formation and extending the fatigue life.

1 Introduction

Cold spray (CS) or gas dynamic cold spray, also known as supersonic particle deposition, is a high-efficient solid-state coating method and powder consolidation process [1]. As seen in Fig. 1, CS uses high-pressure carrier gas (usually nitrogen or helium) heated electrically, to obtain the very high velocity of the metal powders passing through a de Laval nozzle above the critical velocity to adhere particles. The most predominant bonding mechanism in CS is considered to “adiabatic shear instability”. When the particles moving at the critical velocity to impact the substrate, a strong pressure field in spherical shape propagates at the interface of the substrate and particle at the point of contact. Because of the pressure field, shear load is produced which causes plastic deformation of the material laterally and creates localized shear straining. The shear straining under critical circumstances exhibits adiabatic shear instability results thermal softening. The adiabatic shear instability forms a viscous flow at outward direction leading to the temperatures near to material’s melting temperature, which basically forms a bonding between the coating and substrate [2, 3]. So, a combination of mechanical interlocking along with the metallurgical bonding creates recrystallization at high strained coating/substrate interfaces is the main bonding mechanism in CS [4]. This system shows significant benefits over other coating systems. It can form mixtures of metallic and non-metallic particulates to create a

coating. This is suitable for processing of heat sensitive materials as there would be very less heat input with no “heat-affected zone”. Most importantly, CS coatings shows low porosity with high deposition efficiency [5, 6]. The CS process can be applied to dimensional restoration, coatings to improve the corrosion and wear-resistant, and repairing of structural components [7].

Fig. 1. A schematic illustration shows the cold spray system to deposit powder using Supersonic Spray Technology (SST).

Magnesium (Mg) alloys are the lightest metallic material, which is attractive to automotive, aerospace and electronics for light weighting trades [8-11]. Mg alloys are positioned in the anodic region of galvanic

* Corresponding author: hjahed@uwaterloo.ca

series. In general, the least noble of metals are typically corroding in the galvanic cell. Thus, Mg alloys are susceptible to form oxides, chlorides, and sulfides when in the harsh environment which limits its extensive applications. Studies reported that the fatigue and corrosion properties of Mg alloys can be improved by surface modification in CS method [12-18] by employing different coating materials, which has higher strength and better corrosion resistance. Aluminum and its alloys (AA) have shown to be an excellent coating material for coating the surface of the Mg alloy parts [19]. When AA comes in contact with air a thin aluminum oxide layer is made which protects the substrate from penetration of the oxygen which in turn results better oxidation resistance. A number of research work has been reported on the surface modification of Mg alloy substrate in CS process using AA as coating materials [12-18]. Tao et al. [20, 21] investigated the corrosion behaviour of AZ91D Mg alloy and concluded that the main cause for Mg shortening is its corrosion fatigue life. They concluded that the corrosion performance of the AZ91D can be significantly enhanced by surface modification in CS process using pure aluminium [20, 21]. Diab et al. [13] studied the fatigue behavior of pure aluminium coatings on extruded AZ31B Mg alloy in corrosive environment and reported a marginal improvement in fatigue performance. Other research also confirmed that the surface modification of extruded AZ31B alloy in CS process using pure Al increased the fatigue strength by 9% [22]. It is seen that surface modification in CS process using pure aluminium cannot meet the industrial requirements. Our recent studies [10] established that the fatigue performance of the cast AZ31B Mg alloys in different environmental conditions increased up to 25% by depositing AA7075 powder. However, it was seen that the CS sample tested in corrosion environment exhibited localized corrosion due to the presence of residual stress, which caused the premature failure results shorter the fatigue life. In practice, most of the case, the automotive parts are coated with inorganic top coat, which basically improve the corrosion resistance of the outer surface. However, there was no study on the effect of surface modification in CS process with addition of top-coat on the fatigue performance of the cast AZ31B Mg alloy. Therefore, in the present study, the feasibility of CS process on the surface modification to improve the corrosion fatigue performance was investigated. Here, the corrosion fatigue performance of the CS samples were investigated for different environmental conditions and the obtained results were discussed.

2 Experimental Details

In this study, the surface of the as-cast AZ31B Mg alloy was modified using powder of AA-based wrought AA7075 having higher strength and longer fatigue life than the AZ31B. As seen in Fig. 2, the shape of particles of the powder was spherical with an average size of 34 μm . The surface modification was performed at the Fatigue and Stress Analysis laboratory of the University

of Waterloo, Waterloo, Canada, using the Supersonic Spray Technologies (SST) Series P CS system manufactured by Centerline Ltd. First, dog bone shape fatigue samples were extracted from a 300-mm diameter billet of as-cast AZ31B following ASTM: E8/E8M-11. Then, the sample surfaces were polished up to 600 grid SiC emery paper and rinse with acetone. Details about the coating parameters can be seen in [10]. As a top-coat, zinc phosphate was employed in e-painting process on the surface of the uncoated AZ31B and the sample coated with AA 7075.

Fig. 2. SEM image shows the morphology of the AA7075 powder used as coating material.

Coated dog-bone shape specimens with a gauge length of 25 mm and a diameter of 7 mm were used for tensile and fatigue tests following ASTM E8. The tensile tests were performed on the coated and uncoated samples. A fully reversed ($R=-1$) load-controlled four-point bending fatigue tests were conducted using an Instron RR Moore machine. In this study, four types of samples were used for fatigue test. As seen in Fig. 3, they are: (1) uncoated, (2) e-painted only, (3) CS AA7075 coated only, and (4) CS AA7075 followed by e-painted (CS+EP). The tests were conducted at different stress amplitudes between 40 to 145 MPa to cover low and high cycle regime.

Fig. 3. Schematic of AZ31B fatigue samples used in this study: (a) uncoated, (b) e-paint, (c) coated with AA7075 (CS), and (d) coated with AA7075 followed by e-paint

A custom-made chamber was installed to the fatigue machine to perform the corrosion fatigue test (Fig. 4). From the top of the chamber, a pipe was inserted to flow the 3.5% NaCl solution onto the specimen gauge section. A constant flow rate of 40 ml/min during testing was maintained to make a continuous film of solution on sample. For all fatigue tests, a constant frequency of 30 Hz was maintained, and tests were stopped when samples break into two parts or reached at 10^7 cycles, which is considered as a run-out. At least two samples were tested for each condition. The tensile and fatigue fracture samples were then characterized using SEM.

Fig. 4. An experimental setup for corrosion-fatigue tests showing the NaCl solution chamber around the specimen.

3 Results and Discussion

3.1 Coating quality

Fig. 5 shows the obtained topography of the AZ31B substrate and the AA7075 coated sample in as-deposited and after grinding conditions. The root means square average roughness (R_a) values as typical surface characteristics measurement is considered as surface roughness. The measured R_a of the substrate (Fig. 5a) is much lower than the coated sample (Fig. 5b, c). The measured R_a value for the substrate is $0.37 \mu\text{m}$. Compared to the substrate, the average R_a value for the AA7075 CS coating surface is 25 times higher and close to $9.61 \mu\text{m}$. The deviation of R_a is due to differences in the step-over distance, the standoff distance, and AA7075 powder size. It is also well known that the CS process exhibits rough surface finish due to powder consolidation process. The surface roughness is a critical issue in fatigue testing. As the R_a value of the as-coated sample was very high, surface roughness was reduced by grinding to 2.61 for tensile and fatigue tests.

3.2 Behavior of coating in tensile loading

The engineering stress-engineering strain plot of the surface modified, and unmodified conditions tested at room temperature is shown in Fig. 6. In general, the ductility and ultimate strength in both conditions are about to the same. However, a significant increase in yield strength (YS) of the surface modified samples was observed. The obtained results show that the as-cast AZ31B sample has a YS of 87 MPa, an ultimate tensile strength (UTS) of 196 MPa and an elongation of 12% during quasi-static tensile loading as seen in Fig. 6.

Fig. 5. Topography shows the surface morphology of the (a) uncoated AZ31B and (b) AZ31B substrate coated with Al7075 in as-coated condition and (c) after polishing.

In contrast, the surface modified samples show significantly higher YS values of 110 MPa where the coating surface was delaminated and cracked (marked by red arrow). The obtained higher YS was basically due the combination of peening effect, covering of porosity and higher strength of coating materials as mentioned in [7, 23].

Fig. 6. Tensile results showing a comparison between the uncoated and coated AZ31B cast alloy.

There were many studies reported the tensile fracture surface of the AZ31B alloys; e.g., [10]. However, there was no information in the literature on how the CS coating behaves during tensile loading. Thus, in this study, the coating fracture and coating/substrate were investigated using SEM. As seen in the Fig. 7a, the spherical powder was plastically deformed and elongated due to the tensile load. At the same time, a number of secondary cracks in the coatings were identified. This is an indication of strong bonding between the particles.

Fig. 7. SEM micrographs shows tensile fracture surfaces in the AA7075 coating (a) cross-section of the coating and (b) at the coating-substrate interface with corresponding EDX image mapping in (c) and (d).

The SEM image at the interface along with the EDX elemental distribution of the Mg and Al are presented in

Fig. 7 (b-d). It is seen that the concentration of the AA is much higher than the Mg which indicates that the coating was delaminated and separated from the substrate inside the coating. Dayani et al. [7] reported that cast AZ31B coated with AA7075 shows higher bonding strength with cohesion type failure in bonding strength test. However, due to the different Young's modulus of the coating/substrate and higher bonding strength between them, delamination was observed in the coating, which basically led to the failure at slightly lower UTS.

3.3 Behavior of coating on cyclic loading

Fig. 8 illustrated the *S-N* curves of the coated and uncoated samples tested in air and corrosive environment of 3.5% NaCl solution at the different stress amplitudes. For comparison purposes, and to show the improvement in fatigue life in the corrosive environment, some earlier fatigue test results are also added to the figure. In particular, the *S-N* curves from fatigue testing of AZ31B extrusion surface modified with CS coated by pure aluminium tested in similar environmental conditions [13, 22] are also presented in the same figure. Generally, the samples modified with AA7075 alloy coating followed by e-paint obtained longer fatigue life related to the others. The fatigue strength of EP on substrate and CS+EP samples increased significantly under corrosive environment, even higher than the as-coated samples. At the same time, the CS+EP sample shows better fatigue behavior compared to the CS pure-Al on AZ31B extrusion in 3.5% NaCl solution [13, 22].

Fig. 8. A comparison of fatigue life of coated and uncoated AZ31B substrate in different environmental conditions.

Unlike the as-cast and as-coated sample fatigue behavior of EP on cast AZ31B and EP on AA7075 coating exhibits a plateau in *S-N* curve at the stress amplitudes of 50 MPa and 80 MPa, respectively. By contrast, the CS sample displays underprivileged fatigue behavior in the corrosive environment. The shorter fatigue life can be correlated to the presence of residual stress in the CS coating resulting stress corrosion cracking [6, 7]. Alternatively, the improvement of fatigue life in CS+EP can be associated with the corrosion properties. As of our previous study [24], the presence of EP increased the

corrosion resistance significantly. Thus, the CS+EP samples obtained higher corrosion resistance results comparatively a reduction of pit formation which delaying the nucleation of cracks and extending the fatigue life. However, the induced residual stress during surface modification in CS process obtained a positive effect on the fatigue performance enhancement [25]. Compared to the unmodified as-cast and extruded AZ31B samples tested in all conditions, the fatigue performance of the surface modified AZ31B cast is improved remarkably in both, air and corrosive environment.

Fig. 9 illustrates SEM images of the fatigue fracture surfaces of CS and CS+EP samples tested at the stress amplitudes of 100 MPa in overall view and magnified views. It is seen that there was no delamination visible at the interface of the CS sample tested in the air. As seen in the Fig. 9 (a, c), the cracks were nucleated from the casting defects and propagate to the substrate. When the cracks reached at a certain length finally tensile like fracture (Fig. 7a) occurred in the coating. In contrast, the CS+EP, with a life around 3 million cycles, shows localized cavity (yellow arrows in Fig 9d) which permitted the NaCl solution to enter into the cavity and react with the substrate at the interface. Then, the little corrosion pits grow further and created a large cavity which basically causes a delamination of the coating. However, the presence of EP, delaying the formation of pits and prolongs the fatigue life.

Fig. 9. SEM images show the overall (a, b) and magnified views (c, d) of fatigue fracture surfaces for the AZ31B magnesium alloy coated with AA7075 alloy samples tested at total stress amplitudes of 100 MPa in air (a, c) and 3.5% NaCl solution (b, d).

Note: #1 - location of pits to enter solution, #2 – delamination of coating due to corrosion, and #3-crack propagated into the matrix.

The enhancement of fatigue life is related to several factors which includes (i) AA7075 powder high strength, (ii) induced compressive residual stresses and (iii) high bonding strength between coating (AA7075) and

substrate (AZ31B cast). As seen in [26], the AA7075 alloys possess a higher fatigue performance than the as-cast AZ31B. So, the surface cracking in the case of CS samples is deferred until high amount of stresses are imposed. The probability of development of sub-surface cracks in cast AZ31B which rupture at lower stresses is removed in CS samples as any premature cracks in the cast AZ31B were showed not to be progressive cracks. The higher impact velocity and temperature of the carrier gas of CS powder on the substrate induced severe plastic deformation resulting in adiabatic shear instability as discussed earlier [14, 25, 27]. Further, the thermal expansion coefficient (TEC) between coating (AA7075) and substrate (AZ31B) significantly influences the residual stress formation. During the surface modification in CS process, the temperature of the carrier gas was 400°C. The higher value of TEC of coating as compared to substrate creates a higher level of mismatch at the interface which causes a higher magnitude of compressive residual stress. It was shown that the residual stress due to the TEC mismatch, and the high impact of CS particles on the as-cast AZ31B sample was about to -122 MPa in the CS AA7075 coatings [10]. Due to the presence of higher amount of compressive residual stress inhibited or delayed the initiation of cracks results improvement in the fatigue life in CS as-cast AZ31B. Moreover, the surface modification in CS process shielded the materials defects like casting porosity which essentially postponed the nucleation of cracks, resulting in longer fatigue life [10].

4. CONCLUSIONS

In summary, the surface modification in CS process significantly improved the yield strength of cast AZ31B Mg alloy in tensile loading. The fatigue life of the CS AZ31B cast Mg alloy is always higher than the uncoated/ unmodified samples in both, air and corrosive environment. However, only the CS process cannot achieve the infinite fatigue life. An efficient top coating is necessary. Thus, this study advocate that the surface modification with addition of top coating can improve the fatigue life significantly.

The authors would like to gratefully acknowledge the financial support of the Natural Sciences and Engineering Research Council of Canada (NSERC), Automotive Partnership Canada (APC) program with contributions from Multimatic Technical Centre, Ford Motor Company, and Centerline Windsor.

References

1. Moridi, S. M. Hassani-Gangaraj, M. Guagliano, M. Dao, *Surf. Eng.* **30**, 369 (2014).
2. T. Hussain, D. G. McCartney, P. H. Shipway, D. Zhang, *J. Therm. Spray. Techn.* **18**, 364 (2009).
3. T. Schmidt, F. Gärtner, H. Assadi, H. Kreye, *Acta Mater.* **54**, 729 (2006).
4. H. Assadi, F. Gärtner, T. Stoltenhoff, H. Kreye, *Acta Mater.*, **51**, 4379 (2003).

5. S. K. Shaha, B. Marzbanrad, H. Jahed, *TMS 2018 147th Annual Meeting & Exhibition Supplemental Proceedings*, 2018.
6. S. K. Shaha, S. B. Dayani, H. Jahed, *TMS 2018 147th Annual Meeting & Exhibition Supplemental Proceedings*, 2018.
7. S. B. Dayani, S. K. Shaha, R. Ghelichi, J. F. Wang, and H. Jahed, *Surf. Coatings Technol.* **337**, 150 (2018).
8. S. M. H. Karparvarfard, S. K. Shaha, S. B. Behraves, H. Jahed, B. W. Williams, *J. Mater. Sci. Technol.* **33**, 907 (2017).
9. S. M. H. Karparvarfard, S. K. Shaha, A. Hadadzadeh, S. B. Behraves, H. Jahed, M. A. Wells, B. Williams, *Magnesium Technology 2017*.
10. D. Toscano, S. K. Shaha, B. Behraves, H. Jahed, B. Williams, *J. Mater. Eng. Perform.* **26**, 3090 (2017).
11. D. Toscano, S. K. Shaha, B. Behraves, H. Jahed, B. Williams, *Mater. Sci. . Eng. A* **706**, 342 (2017).
12. H. Bu, M. Yandouzi, C. Lu, D. MacDonald, B. Jodoin, *Surf. Coatings Technol.* **207**, 155 (2012).
13. M. Diab, X. Pang, H. Jahed, *Surf. Coatings Technol.* **309**, 423 (2017).
14. R. Ghelichi, D. MacDonald, S. Bagherifard, H. Jahed, M. Guagliano, B. Jodoin, *Acta Mater.* **60**, 6555 (2012).
15. M. R. Rokni, C. A. Widener, G. A. Crawford, M. K. West, *Mater. Sci. Eng. A* **625**, 19 (2015).
16. G. Shayegan, H. Mahmoudi, R. Ghelichi, J. Villafuerte, J. Wang, M. Guagliano, H. Jahed, *Mater. Des.* **60**, 72 (2014).
17. K. Spencer, D. M. Fabijanic, M. X. Zhang, *Surf. Coatings Technol.* **204**, 336 (2009).
18. Q. Wang, D. Qiu, Y. Xiong, N. Birbilis, M. X. Zhang, *Appl. Surf. Sci.* **289**, 366 (2014).
19. S. M. Hassani-Gangaraj, A. Moridi, M. Guagliano, *Surf. Eng.* **31**, 803 (2015).
20. Y. Tao, T. Xiong, C. Sun, L. Kong, X. Cui, T. Li, *Corros. Sci.* **52**, 3191 (2010).
21. Y. Tao, T. Xiong, C. Sun, H. Jin, H. Du, T. Li, *Appl. Surf. Sci.* **256**, 261 (2009).
22. E. Kalatehmollaei, H. Mahmoudi-Asl, H. Jahed, *Int. J. Fatigue* **64**, 33 (2014).
23. B. Marzbanrad, H. Jahed, E. Toyserkani, *Mater. Des.* **138**, 90 (2018).
24. S. K. Shaha, S. B. Dayani, H. Jahed, *Int. J. Fatigue*, (under review), 2018.
25. H. Jahed R. Ghelichi, *Modern Cold Spray : Materials, Process, and Applications* 2015.
26. T. S. Shih, Y.W. Chiu. *Appl. Surf. Sci.* **264**, 410 (2013).
27. R. Ghelichi, S. Bagherifard, D. Mac, M. Brochu, H. Jahed, B. Jodoin and M. Guagliano, *Int. J. Fatigue*, **65**, 51 (2014).