Wear Analysis of DLC Coated Magnesium Composite for Biomedical Application

Michael Sukham, Umasankar V*
School of Mechanical and Building Sciences, VIT University Chennai – 600127
***Corresponding author: E-Mail: umasankar.v@vit.ac.in**

ABSTRACT

Bio medical implants require high strength to mass ratio with good wear and corrosion resistant properties. Magnesium TiO_2 composite was manufactured by extrusion process. The samples are then coated with diamond like carbon (DLC) which is deposited by Plasma Assisted Chemical Vapor Deposition process (PACVD) to provide a uniform coating thickness along with good wear and corrosion resistance. Coating has a micro hardness of 15GPa. Wear properties of DLC coated and uncoated magnesium TiO_2 composite in simulated bio fluid (SBF) ringer solution is being measured to check its compatibility in bio medical applications. In the abrasive test, there was negligible loss in the mass of the coating and wear properties improved with the increase in the TiO_2 content in the composite.

KEY WORDS: DLC coating, TiO_2, Pin on disk test, wear rate, Ringer Solution

1. INTRODUCTION

Magnesium is the lightest metal which shows a high strength to weight ratio, good thermal and electrical conductivity with good shock absorbing properties. The density of Mg and its alloys are around 1.74 g/cm^3 at 20ºC, which is 1.6 times less dense than aluminium. The density of natural bone ranges from 1.8 g/cm^3 to 2.1 g/cm^3 which is slightly higher than that of magnesium and its alloys. The elastic moduli of pure Mg is 45GPa and for human bone it varies between 40 and 57GPa. Due to its close similarity and also Mg being a bio degradable metal with appropriate mechanical properties can be used in body implants. From the study by Choi (2007), it is known that the incorporation of passive oxide forming metal in the coating will result in the increase in the resistance to corrosion. Also from the study by Pagnoux (2014), shows that DLC coatings are very sensitive to the roughness of the counter body and textured DLC coatings are more likely to shows better tribological behavior than non-textured coating. However the main disadvantage of magnesium and its alloys is that they are prone to corrosion which is due to the fact that magnesium is one of the most electrochemically active metal. Another issue is the formation of hydrogen during corrosion.

However these can be avoided by using surface treatments or foam coatings. So Mg and its alloys are coated with various materials to overcome its weakness. DLC or diamond like carbon coating exhibit low friction, corrosion resistance, wear resistance and bio compatibility end as well as the uncoated end will both be tested for wear properties using pin on disk test and compared. The diameter used will be 30mm. A load of 9.81N with the pin rotating at 100rpm is applied. The environment used will be ringer solution which is a simulated body fluid. The test will be carried out for 30 minutes. The data obtained from this experiment will be compared using line graph of track distance vs. wear rate. This will give us an idea as to how the coating influences the wear in the lubricated condition.

2. EXPERIMENTALPROCEDURE

Wear characterization of Mg, Mg+1.98%TiO_2 and Mg+2.5%$TiO_2$is carried out using pin on disk test (TR-20LE-PHM-250). The software used on the test rig was Win DUCOM 2010. Simulated body fluid, ringer solution is prepared for the testing. A standard isotonic ringer solution is prepared by dissolving 6.5g NaCl, 0.42g KCl, 0.25g $CaCl_2$ and 0.2g of sodium bicarbonate in one liter of distilled water. The pH value recorded was 6.85. The wear test was carried out in partial lubrication of ringer solution by providing the solution drop by drop at the pin during the test. The disk material used was EN31 which is high carbon high chromium steel. The disk diameter used is 30mm with 100rpm. The temperature and pressure under which the experiment was performed was room temperature and atmospheric pressure. The load applied was 9.81N and the test was carried out for a duration of 30 minutes. After the pin on disk test is done on both the coated and uncoated tips, the wear data will be compared by plotting the wear rate of the samples with time. The coated and uncoated are compared separately to get an idea about the influence of TiO_2 content in both samples in influencing the wear rate. The graph is plotted with x-axis as time and y-axis as wear. Surface characterization is performed using Field emission scanning electron microscopy (FESEM) and energy dispersive X-ray spectroscopy (EDS).

3. RESULTS AND DISCUSSION

The surface characterization is obtained using Field emission scanning electron microscopy (FESEM) with energy dispersive X-ray spectroscopy (EDS). This will allow us to see the wear characteristics of the sample with the disk material along with EDS for chemical characterization of the sample. FESEM has been taken for both sample of coated and uncoated Mg2.5%TiO_2. EDS has been taken only for the coated sample. Wear rate of the samples are plotted against time. The uncoated and coated samples are compared to study the effect of TiO_2 content in the composite coating and the wear resistance of the coating itself. The red line denotes the wear rate of the DLC coated sample and the blue represents the uncoated base material.

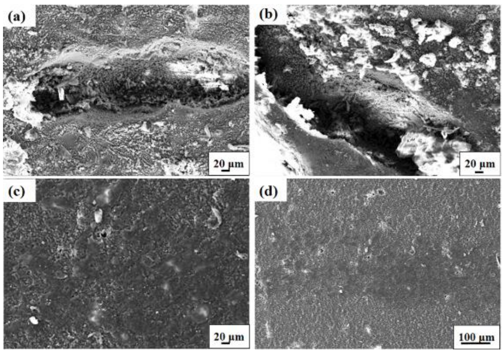

Figure.1. (a), (b) FESEM image of uncoated Mg 2.5%TiO_2. (c), (d) FESEM image of coated DLC Mg 2.5%TiO_2

The FESEM images shows that wear is clearly visible in the case of uncoated sample. Magnesium has very low wear and corrosion resistance. Figure.1. (a) and (b) shows the abrassion of the disk particles into the sample which is causing the wear.Since the test conditions are sliding friction, the disk material which is high carbon high chromium steel, gets impinged into the sample as shown in figure.1. (b) or damages the surface resulting in the removal of the sample material as shown in figure.1. (a). The formation of wear contours leaves the sample open to atmospheric conditions which can furthur lead to corrosion and other reactions leaving it unsuitable for bio medical use. However in the case of the coated sample i.e. Figure.1. (c) and (d), there is slight decolourisation in the coated area. DLC or diamond like carbon coating exhibit low friction, corrosion resistance and wear resistance which is shown by the images. As compared to the uncoated samples, DLC has better wear resistance than the uncoated sample.

Spectrum 1:

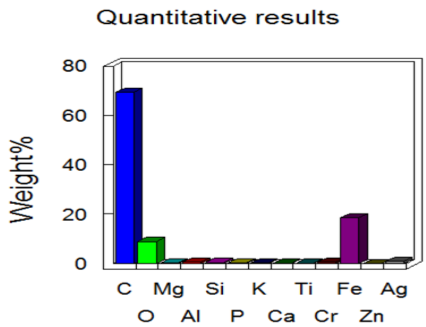

Figure.2. EDS results of DLC coated Mg-2.5%TiO_2 (performed on figure.1. (d) Spectrum 1)

The EDS results show a high percentage of carbon which is due to the DLC coating. There is presence of Fe particles in the sample. Since no visible wear is detected on the sample, the presence of Fe particles is due to the wear in the counter body i.e. disk and the disk particles after getting worn gets impinged into the sample surface.

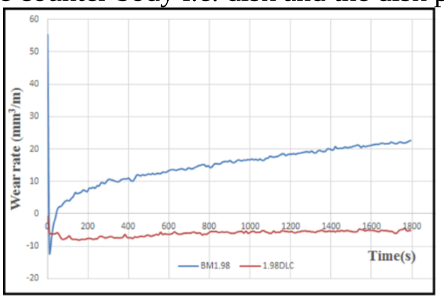

Figure.3. Wear comparison DLC coated and uncoated Mg sample

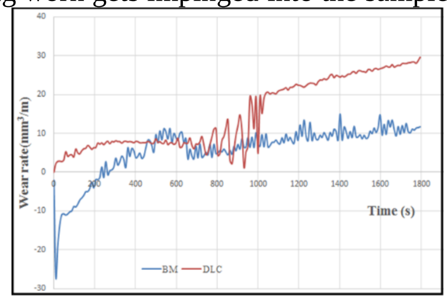

Figure.4. Wear rate comparison of Mg 1.98%TiO_2 DLC coated and uncoated sample

In case of the composite with DLC coating and no TiO_2 content, there is a critical failure in the coating which is shown by the sudden rise in the wear rate from 800secs-1000secs. The base material shows a lesser rise in the wear rate showing there is gradual wear. The critical wear of the coated sample shows the delamination and failure of the coating under the wear conditions which is not desirable in any circumstance. Since the medium used was corrosive in nature and thin DLCs are known to have lesser resistance to wear and scratches, this may have resulted in the critical failure of the coating.

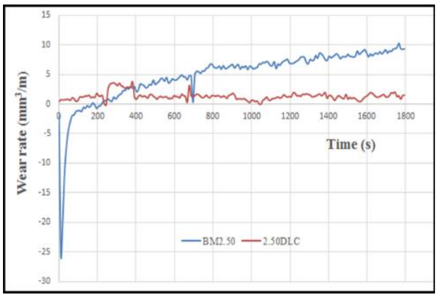

Figure.5. Wear rate comparison of Mg 2.5%TiO_2 DLC coated and uncoated sample

In case of the composite with 1.98% TiO_2 content, there is varied wear rate between the coated and uncoated sample. Although the wear rate is low in the coated sample, the uncoated sample shows a very high wear rate. Due to the varied wear rate, this coating sample when it fails will exhibit high wear rate from the exposed part, thus making it unsuitable for use. However there are no signs of critical failure as seen in figure 1.

The wear rate for the coated and uncoated Mg 2.5%TiO_2 shows only gradual wear with no critical failure. The coated sample shows a uniform wear and also the uncoated sample shows a little higher wear from the coated sample. Initial high wear rates may be due to the non-uniformity in coating surface. The base material have almost same wear rates however the wear rates are decreased with the content of TiO_2 in the composite. So even if the coating failure occurs, the uncoated area won't undergo severe wear as in the cases above which makes it very suitable for bio applications.

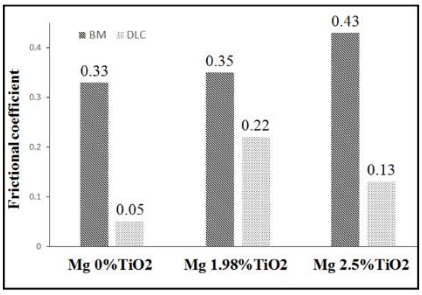

Figure.6. Friction coefficient comparison of base material (BM) with coated samples (DLC)

Average friction coefficient is calculated from the frictional force obtained from Pin on Disk test. There is gradual rise in the value in case of base material with the increase in TiO_2. The graphitization which occurs during friction when the sp3 structure converted into the sp2 structure due to a friction-induced graphitization mechanism. The transfer layer formed on a counter face through the graphitization process provides low shear strength between hexagonal planes in the graphite structure allowing for the possibility of reducing friction coefficient which is why the friction coefficient of DLC coated samples are much lower than the uncoated samples.

Mg DLC coating with no TiO_2 (in figure.3) shows critical wear rate which may be due to adhesion problem and the bio fluid environment in which the test was conducted. Mg DLC coating with 1.98% TiO_2 (figure.4) shows good wear resistance. However the uncoated sample shows poor wear resistance relatively to the coated sample. The Mg with 2.50%TiO_2 (figure.5) shows better wear resistance in both the coated and uncoated samples. There is only a slight increase in the wear rate for the uncoated sample for 2.5%TiO_2which makes it safe even after the coating failure. There were no sign of corrosion in the coated samples which shows that DLC is a viable option of coating for body implants as it is resistant to the simulated body fluid. Further tests can be performed to solidify the claim.

4. CONCLUSION

Medical applications such as implants require light weight bio compatible materials. Magnesium has low density than aluminium however its use is limited because of low friction and wear resistance. TiO_2 content in the composite increases the wear resistance of the sample in the expense of increase in the friction coefficient. Diamond like carbon coating (DLC) overcomes this problem and provides a good wear resistivity to the magnesium TiO_2 alloy without further increasing the density and friction coefficient, thus retaining the light weight as well as reducing the sliding friction stress. Also magnesium is bio compatible which makes it a viable option for bio medical applications. Thus, DLC improves the wear resistance of the Mg TiO_2 alloy without increasing the friction coefficient.

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