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Comparative study of the wear resistance of Al_2O_3-coated MA956 superalloy(•)

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Abstract Preoxidation of the MA956 superalloy, proposed as a prosthetic biomaterial, generates a compact and inert α-alumina surface layer. The aim of this study is to assess the wear resistance provided by this alumina layer on the MA956 alloy in comparison with the same coated alloy and versus high density polyethylene. Comparative wear tests were carried out in both dry and wet conditions using the couples MA956/MA956, MA956/UHMWPE (ultra high molecular weight polyethylene) and 316LVM/UHMWPE. The results corresponding to the couples MA956/MA956, with and without alumina layer, show that the load has more significant effect than the rotation speed on the weight loss and on the roughness. On the other hand the alumina surface layer provides a clear wear protection. The weight losses of the MA956 specimen in this couple are ten times lower when testing under wet conditions than under dry conditions. The MA956/UHMWPE couple behaves much better than the 316LVM/UHMWPE, as it presents the lowest values of friction coefficient and weight losses of the MA956 specimen. These are only detectable after 70,000 cycles under a 70 MPa contact pressure. This couple offers the best guarantee of a prolonged service life for articulated parts in a prosthesis.

Keywords Wear. MA956. Alumina coating. UHMWPE.

Estudio comparativo de la resistencia al desgaste de la superaleación MA956 recubierta de Al_2O_3

Resumen La superaleación MA956, que se ha propuesto como biomaterial para prótesis osteoarticulares genera, mediante un adecuado tratamiento de preoxidación, una capa superficial, compacta e inerte, de alúmina alfa. El objetivo del presente trabajo es el estudio de la resistencia al desgaste de esta capa de alúmina, cuando se encuentra en contacto con la propia aleación recubierta y con polietileno de alta densidad. Se realizaron ensayos comparativos de desgaste, tanto en seco como en húmedo, con los pares MA956/MA956, MA956/UHMWPE (polietileno de muy elevado peso molecular) y 316LVM/UHMWPE. Los resultados obtenidos con el par MA956/MA956, con y sin capa de alúmina, indican que la carga es el factor de mayor importancia en cuanto a los efectos de pérdida de peso y rugosidad. Por otra parte, la presencia de la capa de alúmina proporciona una clara protección frente al desgaste. Las pérdidas de peso de la MA956 resultan ser unas diez veces menores en el ensayo en húmedo que en el ensayo en seco. El par MA956/UHMWPE presenta los menores valores del coeficiente de fricción y unas pérdidas de peso de la probeta metálica, apenas detectables al cabo de 70.0000 ciclos de carga, con una presión de contacto de 70 MPa. Este par ofrece la mejor garantía para un prolongado servicio de los elementos articulares de una prótesis.

1. INTRODUCTION

One of the essential requirements which must be satisfied by a biomaterial for arthroplasty, besides biocompatibility and chemical inertia, is that of adequate resistance to mechanical loading, to which joint replacement prostheses are subjected after their implantation. In particular, there is increasing concern for improving the wear resistance of these materials. The production of wear particles on bearing surfaces has recently been considered to be of major importance in aseptic

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loosening of the prostheses in total arthroplasty[1-3]. These released particles can lead to a periprosthetic tissue reaction and bone lysis[4 and 5]. Much attention is being paid to the study of the pathogenic mechanisms of the loosening process[6 and 7], as well as to the study of wear phenomena in joint implants and of the different materials and coatings which can be used for bearing surfaces[8 and 9]. The interest in reducing as far as possible wear debris in prostheses constitutes a very important objective in this field, not only because it means prolonging the useful lifetime of implants from a mechanical point of view, but also because it reduces the systemic and local biological risks associated with the detachment of foreign particles inside the human body.

The material couples most commonly used for the bearing surfaces of ball heads and acetabular cups in hip prostheses include Al_2O_3/Al_2O_3, Al_2O_3/polyethylene, metal/polyethylene and metal/metal[10]. A ceramic/polyethylene combination reduces by half the total amount of wear produced by a metal/polyethylene couple[11]. The main advantage of ceramics is their greater wettability, which causes a drastic reduction of the friction coefficient and thus less adhesive wear of polyethylene. Another of the advantages presented by ceramics is their capacity to maintain surfaces with extremely low roughness during very long service times. In metal/polyethylene couples, passive oxide layers on the metallic bearing surface tend to deteriorate and regenerate continuously as a consequence of the friction. This fact reduces the metallic surface wettability, increases its roughness, and leads to the appearance of third-body abrasive wear, due to the oxide particles. Therefore, in addition to adhesive wear, polyethylene wear is greatly increased.

The MA956 superalloy has been proposed as a possible biomaterial for joint prostheses in view of its excellent corrosion resistance characteristics[12 and 13] achieved after a treatment at high temperatures. This treatment gives it a greater chemical inertia than that found in conventional materials currently in use[14]. Since one of the most pursued goals in the field of arthroplasty is to improve wear resistance, this work is aimed at assessing the wear resistance provided by the alumina layer generated[15] on the MA956 alloy versus the same coated alloy. In addition it was considered appropriate to compare the wear behaviour of the MA956/UHMWPE and 316LVM/UHMWPE couples in view of the common usage of polyethylene acetabular cups in hip prosthesis prostheses.

2. MATERIALS AND METHODS

The materials used in this work were MA956 superalloy, AISI 316LVM stainless steel, and an ultrahigh molecular weight polyethylene, UHMWPE.

The MA956 superalloy was supplied by Inco Alloys Limited of Hereford (UK) and had a nominal composition of Fe-20Cr-5Al-0.5Ti-0.5Y_2O_3-0.01C (wt %). This is therefore a ferritic stainless steel, strengthened by oxide dispersion. For the MA956/MA956 coupling, the material used was treated at 1100 °C for 100 h. This treatment generates an alumina layer by oxidation. The heat treated alloy has a yield strength of 810 MPa, a tensile strength of 990 MPa and a total elongation of 21 %.

The stainless steel AISI 316LVM was used as reference material and was supplied by Stahlwerk Ergste of Schwerte, Germany, in the form of a rod prepared by rolling and had a composition Fe-20Cr-14Ni-3Mo-2Mn-0.03C (wt %).

The UHMWPE used in the wear tests is the type regularly employed for acetabular cups in hip prostheses and was supplied by a manufacturer of joint prostheses. It has a density of 0.93 g/cm^3 and a yield strength of 23 MPa.

The comparative wear tests were carried out in an Amsler Universal machine which allows selection of the appropriate load and rotation speed. For these tests, two cylindrical specimens were used with external diameter of 35 mm and 10 mm width (Fig. 1). The external surface of the samples was ground before testing. The samples support a load P and undergo sliding and rolling. Sliding is caused by the 10 % difference between the rotation speeds of the two specimens. The contact area was determined in each case using the Hertz formula as a function of the load and external diameter.

The tests were carried out using three different couples:

MA956/MA956
MA956/UHMWPE
316LVM/UHMWPE

The MA956 alloy in the first couple was tested with and without alumina surface coating. In order to guarantee the same microstructural state and that any difference in behaviour could be attributed exclusively to the surface layer, all the

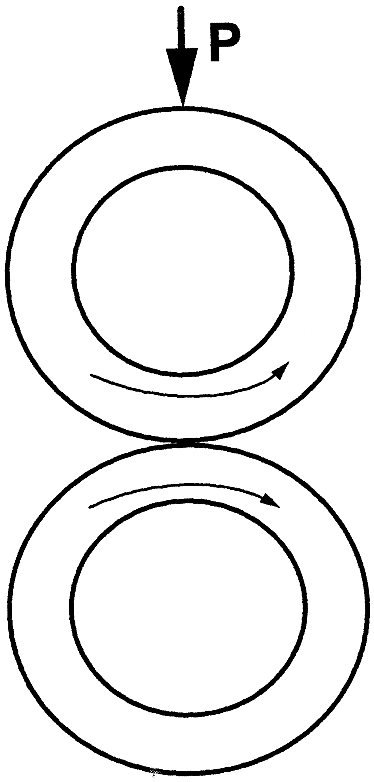

Figure 1. Wear tests arrangement.

Figura 1. Configuración del ensayo de desgaste.

samples used were treated under the same conditions and the alumina coating was mechanically removed in one of the samples of the couple.

In the MA956/UHMWPE couple the MA956 specimens always incorporated the alumina layer. Given that the wear behaviour of these first two couples was the main objective of this study, the corresponding tests were carried out with three load levels, namely 19, 37 and 56 N, and three rotation speed levels: 8, 16 and 24 rpm

For the 316LVM/UHMWPE couple the tests were carried out using only a rotation speed of 24 rpm, at the three load levels indicated above.

All the wear test were carried out both in dry conditions and after wetting the contact area with Hank's solution (Table I), in order to simulate the effect of organic liquids. In most cases it was considered sufficient to limit the tests to 85,000 cycles, bearing in mind that the loads used were relatively high in comparison with those normally found in service conditions.

Prior to the performance of each test the specimens were cleaned, weighed and their initial surface roughness measured. For the determination of the standard roughness parameters a Mitutoyo Surftest 401 measuring unit was used. The cleaning and weight loss monitoring operations were repeated all throughout the partial stages until the final number of cycles was reached. The continuous recording of the torque during the test allowed the calculation of the static and dynamic friction coefficients in each experiment. The surface roughness of the two specimens of each tested couple was again measured at the end of each test.

3. RESULTS AND DISCUSSION

Figure 2 shows a cross-sectional micrograph of the α-Al_2O_3 surface layer cross section in the MA956 alloy. The oxide layer formed by oxidation in air at 1100 °C for 100 h had a thickness of the order of approximately 4 μm. The alumina surface layer is

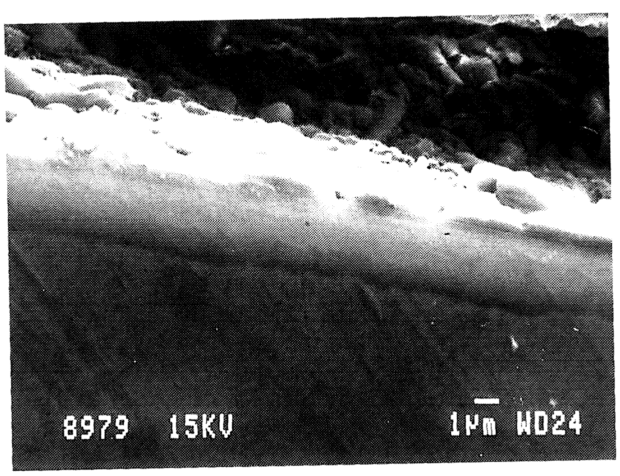

Figure 2. Secondary electron image of the layer cross section of MA956 superalloy after 100 h at 1100 °C.

Figura 2. Imagen de la sección transversal de la superaleación MA956 tratada a1100 °C durante 100 h.

Table I. Hank's solution composition (g/l)

Tabla I. Composición de la solución de Hank (g/l)

$CaCl_2$	KCl	KH_2PO_4	$MgSO_4$	NaCl	$NaHPO_4$	$NaHCO_3$	d-glucose
0.185	0.40	0.06	0.0976	8.00	0.04788	0.35	1.00

dense, compact, adherent and shows high chemical stability.

3.1. Dry wear testing of the MA956/MA956 couple

Tests were initially carried out using coated MA956 alloy couples. Table II shows the results obtained in these tests for the different load levels and rotation speeds. This table includes measured values for the weight loss, friction coefficient and standard roughness parameter, R_a. To evaluate the effect of the load and the sliding speed on the results obtained, a study was made using variance analysis techniques. In this study these two testing conditions were considered as factors, while consideration was made separately of either the weight loss, friction coefficient or standard roughness parameter R_a as single response variables. Tables III and IV show the average values obtained, indicating the corresponding confidence level values of the effects of the two factors, load and speed respectively, on each of the three aforementioned dependent variables.

Table III shows that the load has a considerable effect on weight loss, and that the statistical confidence level of this effect is very low. The load is also seen to have a clear effect on the roughness value, with a statistical confidence level, α, also lower than 0.01. The friction coefficient, however, as was to be expected, does not show variation with the load level.

Table II. Results obtained from wear tests in the MA956 coated with alumina

Tabla II. Resultados de los ensayos de desgaste de la aleación MA956 con una capa superficial de alúmina

Load N	Rotation speed rpm	Weight loss mg	Friction coeff. μ	Roughness, R_a μm
56	8	95.0	0.18	2.10
56	16	108.0	0.36	2.50
56	24	54.0	0.26	3.10
37	8	69.0	0.22	1.80
37	16	71.0	0.24	2.65
37	16	41.0	0.21	2.40
37	16	48.0	0.20	2.55
37	24	40.0	0.19	2.45
19	8	10.0	0.20	1.30
19	16	34.0	0.40	1.50
19	24	8.0	0.31	1.45

Table III. Weight loss, friction coefficient and roughness mean values, for MA956 with surface alumina layer, as a function of the load

Tabla III. Pérdidas de peso, coeficientes de fricción y valores medios de rugosidad de la aleación MA956 recubierta de alúmina, en función de la carga

Load N	Weight loss mg	Friction coeff. μ	Roughness, R_a μm
19	17,33	0,30	1,42
37	49,84	0,19	2,32
57	85,67	0,27	2,57
Confidence Level, α	<0,01	0,07	<0,01

Table IV. Weight loss, friction coefficient and roughness mean values, for MA956 with surface alumina layer, as a function of the rotation speed

Tabla IV. Pérdidas de peso, coeficientes de fricción y valores medios de rugosidad de la aleación MA956 recubierta de alúmina, en función de la velocidad de giro

Rotation speed rpm	Weight loss mg	Friction coeff. μ	Roughness, R_a μm
8	58,00	0,20	1,73
16	60,84	0,31	2,23
24	34,00	0,25	2,33
Confidence Level, α	0,14	0,09	0,04

On the other hand, there is no appreciable variation of the weight loss or friction coefficient with rotation speed as seen in table IV. The surface roughness, however, shows a slight increase with increasing speed level.

In order to obtain an overview of the wear process of the alumina coated MA956 alloy a three-dimensional wear map has been elaborated, in which the weight loss is represented as a function of the load and sliding speed (Fig. 3). In this figure we see that the increase in weight loss occurs systematically with increasing load for all the speeds. However, a maximum wear value is observed in the 16 rpm zone for all the load levels. This zone could correspond to a smooth-severe-smooth transition of the wear process. It can also be noted that the effect of the load variation has a

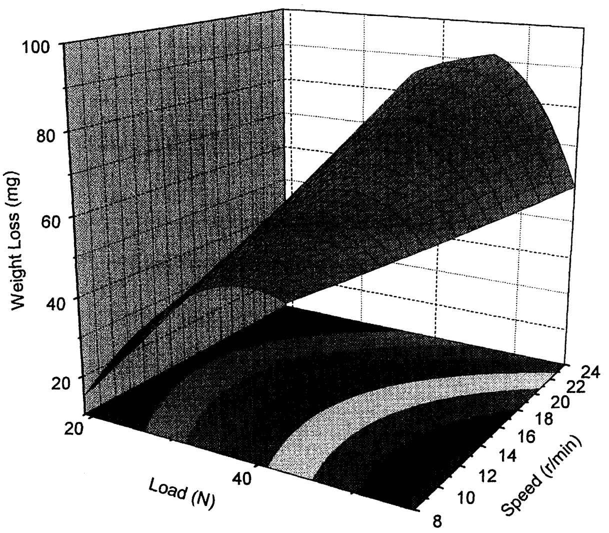

Figure 3. Three-dimensional wear map of the Al_2O_3-coated MA956 superalloy as a function of the load and sliding speed.

Figura 3. Mapa tridimensional de desgaste de la superaleación MA956 recubierta de Al_2O_3 en función de la carga y de la velocidad de giro.

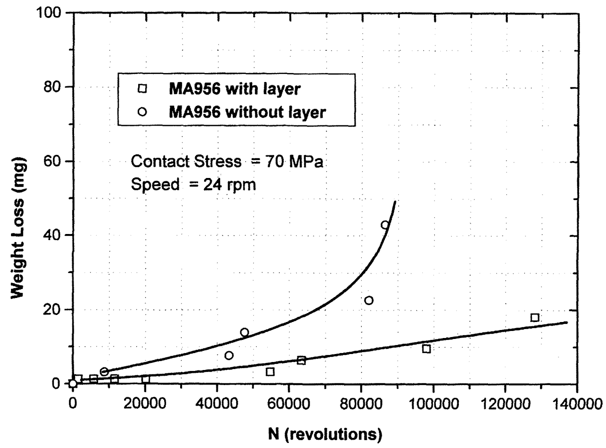

Figure 4. Comparative wear tests of the MA956 superalloy, with and without alumina layer.

Figura 4. Ensayos comparativos de desgaste de la superaleación MA956, con y sin capa de alúmina.

qualitatively greater importance than that due to the speed variation. When assessing the tribological behaviour of this couple we should consider that the loads, of 19, 37 and 56 N, used for the tests correspond to contact pressures of 70, 95 and 120 MPa, which are much greater than the service pressures of 5 MPa.

To determine the effect of the alumina layer generated by the oxidation treatment on the wear resistance of the alloy, comparative wear tests were carried out using MA956 specimens with and without alumina coating but treated under the same conditions such that possible differences in wear behaviour could be attributed exclusively to the surface layer. The tests were carried out with a load of 19 N and a rotation speed of 24 rpm in both cases. Figure 4 displays the results obtained. In this figure we notice that the alumina surface layer provides a clear wear protection for the MA956 alloy. Specifically, after 86,400 cycles there is a sudden increase in the slope of the weight loss curve in the case of the uncoated material, while the losses measured for the coated material show a practically linear increase even above 120,000 cycles.

3.2. Dry wear testing for the comparison of the MA956/MA956 and MA956/UHMWPE couples

To establish this comparison tests were carried out using polyethylene specimens in contact with treated MA956 specimens coated with the alumina surface layer. The appropriate loads were applied to achieve pressures in the contact area of the MA956/UHMWPE couple equivalent to those obtained in the tests carried out on the MA956/MA956 couple. Bearing in mind the elastic constants of polyethylene, E = 2.55 GPa and ν=0.41, it was calculated that a load of some 345 N was required to reach the pressure of 70 MPa estimated for the MA956/MA956 couple of the previous tests. Figure 5 displays the weight losses obtained with this load for a speed of 24 rpm. In this figure the weight losses have been represented separately for each of the specimens of the

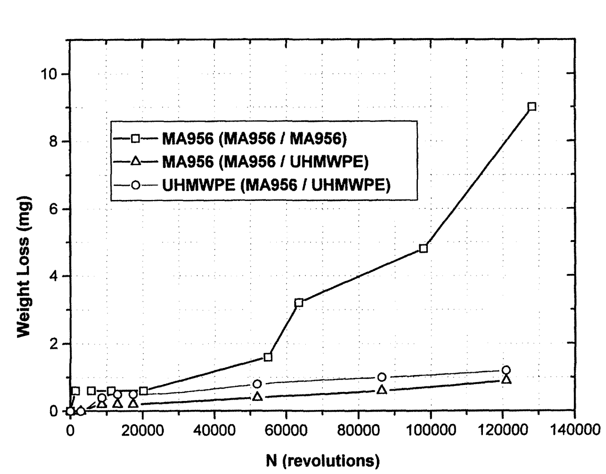

Figure 5. Comparative dry wear tests of the MA956/MA956 and MA956/UHMWPE couples at a pressure corresponding to 70 MPa and a speed of 24 rpm.

Figura 5. Ensayos comparativos de desgaste en seco de los pares MA956/MA956 y MA956/UHMWPE realizados con 70 MPa de presión de contacto a una velocidad de 24 rpm.

MA956/UHMWPE couple and as an average value for both specimens of the MA956/MA956 couple. It can be seen that the weight losses of the alumina coated MA956 specimen in the MA956/UHMWPE couple are somewhat lower than those measured for the corresponding polyethylene specimens. Furthermore, bearing in mind the density ratio between alumina and polyethylene, we estimate that the volume of alumina particles detached represents approximately 25 % of the volume corresponding to polyethylene abrasion particles. On the other hand, when comparing the weight losses of the MA956 specimen tested against a polyethylene specimen with the average weight losses of the MA956/MA956 couple a significant difference is seen even though the contact pressure was the same. In both cases the wear observed in the MA956 specimen remained within the limits of the alumina surface layer (i.e. wear is restricted exclusively to the coating).

3.3. Wet wear testing of the MA956/MA956 and MA956/UHMWPE couples

In these tests a Hank's physiological solution was used as lubricant in the specimens' contact zone. The weight losses obtained in wet tests under the same contact pressure and speed conditions as in the dry experiments, are given in figure 6. When comparing this figure with figure 5 it should be taken into account that the scale has been reduced by approximately one order of magnitude. The weight losses of the MA956 specimens are seen to be about ten times lower than those obtained under dry testing for the MA956/MA956 couple. The weight losses corresponding to the MA956 specimen tested against a polyethylene specimen were also reduced by approximately the same proportion, and when they were low in dry testing, they were only started to be detected after 70,000 cycles in wet testing.

The curve corresponding to the wet wear results of polyethylene presents however very similar values to those seen in figure 5 for the dry tests, and in fact is higher than that corresponding to the MA956 specimens in the MA956/MA956 couple. The apparent insensitivity of the polyethylene wear process to the presence of the Hank's solution can be attributed to the low wettability of this material, in comparison with the interfacial behaviour of the water-alumina system[16].

Figure 7 shows the evolution of the friction coefficients throughout the experiments of the tested couples. The friction coefficient values with the polyethylene specimen are seen to be much lower than those measured for the MA956/MA956 couple, both under dry and wet testing conditions. With regard to the lubricant effect of the physiological solution in the contact area, a very clear reduction is seen in the coefficients measured in wet testing for both couples, although the reduction corresponding to the MA956/MA956 couple is much larger. It should also be noted that in the case of dry testing of the MA956/MA956 couple there is a considerable increase in the friction coefficient with testing time, while in wet testing it remains almost constant until the end of the test.

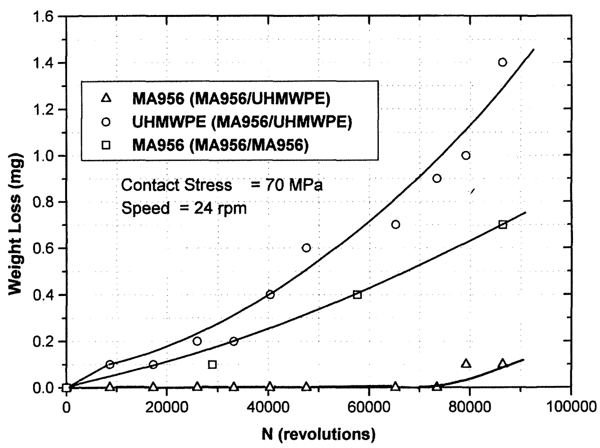

Figure 6. Comparative wet wear tests of the MA956/MA956 and MA956/UHMWPE couples at a pressure corresponding to 70 MPa and a speed of 24 rpm.

Figura 6. Ensayos comparativos de desgaste en húmedo de los pares MA956/MA956 y MA956/UHMWPE realizados con 70 MPa de presión de contacto a una velocidad de 24 rpm.

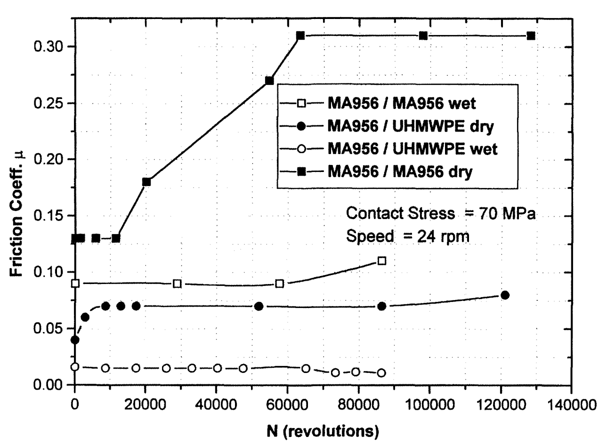

Figure 7. Evolution of the friction coefficients of the tested couples.

Figura 7. Evolución de los coeficientes de rozamiento de los pares ensayados.

This difference is explained by the lower weight losses during wet testing which is a consequence of a reduced friction as the contact surface is in good condition for sliding for longer time.

With regard to the MA956/UHMWPE couple, low values of the friction coefficient are seen even in dry testing, and it is therefore not surprising that this coefficient hardly increases during the testing time, as the low weight loss values seen in figure 5 indicate that little surface deterioration occurs with friction. In wet testing the friction coefficient values measured for this couple are even lower, and no tendency is seen for the curve to increase during the testing time. The low friction coefficient values measured for this couple agree with the data in the literature[17] and confirm that the alumina-polyethylene couple provides excellent friction behaviour.

3.4. Comparative tests in wet conditions with the 316LVM steel/UHMWPE couple

The 316LVM specimens were tested against UHMWPE specimens in the presence of a physiological solution. The total volume losses measured during these tests are shown in figure 8, together with the corresponding values for the MA956/UHMWPE couple. It can clearly be seen that the MA956/UHMWPE couple suffers less wear than the 316LVM/UHMWPE couple for the same load and speed conditions. In both cases most of the volume loss of the couple logically corresponds to the polyethylene specimen.

Finally, figure 9 displays the values of the dimensional wear constant *k*. This is an index of the volume lost per unit of sliding length and per unit of load applied on the contact surface. In this figure we see that the dimensional wear coefficients *k* of the couples which include a polyethylene specimen are similar. However, the coefficient of the MA956/MA956 couple is larger by two orders of magnitude. This can be explained by the fact that in this couple the alumina coating starts to deteriorate when subjected to loads greater than 10 N, which corresponds to a contact pressure of 70 MPa. On the other hand, in the MA956/UHMWPE couple the coating suffers no deterioration under the load of 343 N, which corresponds approximately to the same pressure of 70 MPa, because the friction coefficient is lower. For all of these reasons it can be stated that the MA956/UHMWPE couple presents excellent wear behaviour. Furthermore it should be noted that these wear coefficient values are of the same order as those reported in the literature [18].

The wear parameters obtained in the different tests carried out with the MA956/MA956 and MA956/UHMWPE couples initially proposed for joint prostheses are satisfactory both in dry conditions and in the presence of a physiological solution. However, an appreciable difference is seen in favour of the alloy's behaviour when it operates in contact with polyethylene. The alumina layer can withstand a high number of cycles without deterioration, but due to its low thickness, the best guarantee of a prolonged service

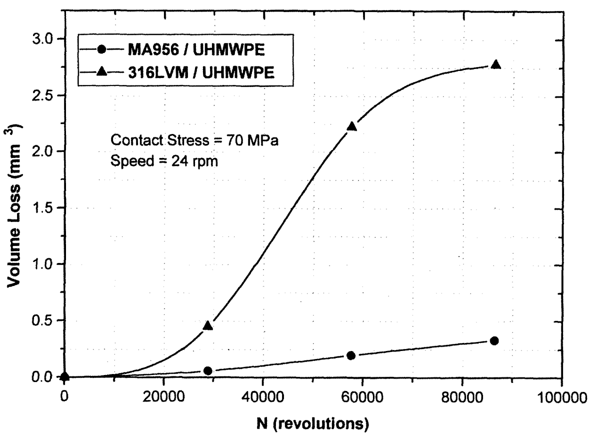

Figure 8. Evolution of the volume loss for the MA956/UHMWPE and 316LVM/UHMWPE couples in wet conditions.

Figura 8. Evolución de las pérdidas de volumen de los pares MA956/UHMWPE y 316LVM/UHMWPE ensayados en húmedo.

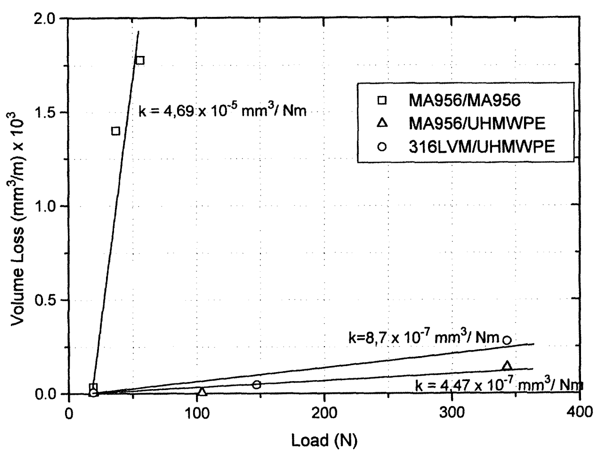

Figure 9. Dimensional wear coefficients *k* for the different couples tested in wet conditions.

Figura 9. Valores de los coeficientes dimensionales de rozamiento k para los distintos pares ensayados en húmedo.

life for articulated parts in a prosthesis would be obtained with the MA956/UHMWPE couple.

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