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ABSTRACT

An experimental comparison has been made between the properties of the surfaces of an $Al_{70}Pd_{21}Mn_9$ quasicrystal and its $Al_{48}Pd_{42}Mn_{10}$ approximant. The $Al_{70}Pd_{21}Mn_9$ sample was a single grain icosahedral quasicrystal cut to expose its five-fold symmetric (000001) surface. The approximant was polycrystalline β-phase $Al_{48}Pd_{42}Mn_{10}$, which has a CsCl-type cubic structure. Surfaces of both were prepared under ultra-high vacuum (UHV) conditions and then used for comparative measurements of their frictional properties and oxidation rates. Both materials are oxidized by reaction with O_2 to form a thin film of aluminum oxide that ultimately passivates their surfaces. The interesting difference between the two is that the rate of oxidation of the approximant is significantly higher than that of the quasicrystal in spite of the fact that the bulk Al concentration of the approximant is lower than that of the quasicrystal. Friction measurements were made under UHV conditions between pairs of quasicrystals and pairs of approximants whose surfaces were either clean or oxidized to varying degrees. The friction between pairs of the approximant surfaces is significantly higher than that measured between the quasicrystal surfaces under all conditions of surface oxidation.

INTRODUCTION

The extraordinary structural properties of quasicrystals have motivated numerous measurements of materials properties that might exhibit anomalous behavior that is a direct result of quasicrystallinity. Two such properties that might, in principle, lead to important commercial and technological applications of these materials are their tribological and corrosion properties. There have been a number of reports of apparently low friction measured on the quasicrystal surfaces and there have also been a number of studies of their oxidation behavior [1-13]. In addition there are other properties of quasicrystals such as high hardness, low thermal conductivity, and interesting electronic properties that might be of practical interest and importance [1, 14-17]. From a materials science perspective, however, the interesting question is whether such materials properties are a direct consequence of quasicrystallinity.

There are several possible origins of the low frictional properties of quasicrystals. One of the most intellectually titillating ideas is that because of their inherent lack of periodicity quasicrystalline surfaces can never come into commensurate contact with one another or with any material having a periodic structure. While the connection between commensurability and friction is far from being clearly resolved at the experimental level there are several theoretical papers that predict such a connection [18,19]. Alternately, if the surfaces of quasicrystals are coated with thin films of contaminants that effectively serve as lubricants, the hardness of the quasicrystals results in low contact area and thus low friction. Hardness may be a result of the quasicrystalline structure and in this case serves as the link between friction and quasicrystallinity [1,14]. A similar idea is that the thin oxide films on air exposed quasicrystal surfaces will serve as lubricants because they are only weakly adherent to the quasicrystal substrate and delaminate under shear [20]. Another suggestion is that the low density of electronic states at the Fermi level can influence friction. Unraveling these competing theories and then making the connection to quasicrystallinity is a difficult experimental challenge that is partly addressed by the results presented in this paper.

There are several reports that quasicrystals exhibit corrosion resistance [21,22]. As in the case of the tribological properties this is, at least in part, a surface phenomenon that will be influenced by surface structure and by surface contamination. The origins of possible corrosion resistance are not clear but are also addressed by the results reported in this paper.

Making the connection between the macroscopic materials properties of quasicrystals and their atomic level structure is a complicated problem. One issue is the need for good experimental measurements under well-defined conditions. This is always an issue in the discussion of tribological phenomena since these are inherently surface related properties and are extremely sensitive to the presence of surface contamination. The second hurdle to any experimental test of the connection between quasicrystallinity and materials properties is that it is not possible to experimentally vary the relevant parameters independently. For example, one cannot vary the structure and composition of the alloys independently. Furthermore, changes in one parameter will cause changes in several properties of the system, some of which may be coupled. Finally, in such problems one is faced with the fact that it is often impossible to change parameters continuously. For example, while the composition of an alloy may be varied continuously its structure is often dictated by a phase diagram that does not allow variation in a continuous manner. As a result of these numerous problems it is difficult to find reliable measurements that allow one to make unambiguous statements about the role of quasicrystallinity in the determination of macroscopic materials properties.

The goal of the measurements described in this paper has been to relate the friction and oxidation properties of quasicrystal surfaces to their structure. Needless to say, despite our best efforts, we do not surmount all the problems mentioned in the previous paragraph. In this investigation we have used two materials. One is the icosahedral quasicrystal, $Al_{70}Pd_{21}Mn_9$, and the other is its β-phase approximant, $Al_{48}Pd_{42}Mn_{10}$, which has a cubic CsCl-type structure. The AlPdMn system is highly complex and several efforts have been made to discern the regions in which quasicrystals and their approximants exist in the phase diagram [23-25]. The stable icosahedral quasicrystalline phase was found to exist at a composition of $Al_{70.3}Pd_{21.4}Mn_8$ [26]. In reality, it exists over a narrow range of compositions of each of its components. Surrounding the quasicrystalline region in the phase diagram are several approximant phases containing various crystalline structures, many of which have been identified [23-27]. The $Al_{48}Pd_{42}Mn_{10}$ approximant used in this study is of an approximant phase that exists over a rather large region of the AlPdMn phase diagram. It is a B2-type approximant phase (usually named β-phase) having a cubic structure and it has a coherent orientation relationship with the quasicrystal as do other frequently encountered approximant phases [28]. In summary, while we have kept the alloy composition as close to that of the quasicrystal as possible the approximant has a periodic rather than quasicrystalline structure.

One of the virtues of the measurements reported in the paper is that they are performed under the well-defined and controlled conditions of ultra-high vacuum (UHV). In all cases the surfaces of the quasicrystal and the approximant alloy have been prepared and cleaned using state-of-the-art surface science methods. Furthermore, the measurements of friction and of surface oxidation have all been performed in UHV without exposure of the surfaces to any form of contamination. The results of these measurements reveal that the friction between the approximant's surfaces is always higher than that measured between the quasicrystal surfaces. Furthermore, the oxidation rates of the approximant alloy are significantly higher than those of the quasicrystal. Although the cause of these differences is not fully understood the results of these measurements serve as reliable starting points for discussion of quasicrystal friction and oxidation.

EXPERIMENTAL

All the experiments were performed in an UHV chamber equipped with a variety of types of instrumentation for surface preparation and surface analysis. In addition the chamber was equipped with a unique device for measurement of friction between pairs of well-defined surfaces, both of which have been subjected to the same preparation and analysis procedures. The original work with the $Al_{70}Pd_{21}Mn_9$ quasicrystal surfaces has been described in another paper which includes a fairly detailed description of the apparatus [29]. Briefly, it is an UHV chamber pumped into the low 10^{-10} Torr pressure range using an ion pump and a titanium sublimation pump. The chamber contains two samples at any one time in order to allow the measurement of friction between them. Both can be heated and cooled over the temperature range 100K to 1000K in order to allow cleaning. The sample surfaces can also be Ar^+ ion sputtered and then analyzed using Auger electron spectroscopy (AES). For the prior work using the $Al_{70}Pd_{21}Mn_9$ quasicrystals the chamber was fitted with a low energy electron diffraction (LEED) optics which was used for the AES measurements and also allowed examination of the sample surface using LEED. For the measurements using the $Al_{48}Pd_{42}Mn_{10}$ approximant the chamber was modified in order to replace the LEED optics with a hemispherical electron energy analyzer and a small spot electron gun for AES.

The samples were made at the Ames Laboratory and were roughly 1 cm^2 in area and about 1 mm thick. The $Al_{70}Pd_{21}Mn_9$ quasicrystals were single grains oriented to expose the five-fold rotation axis normal to the surface. The $Al_{48}Pd_{42}Mn_{10}$ approximants were polycrystalline. Prior to any measurements the samples were subjected to extensive cleaning by Ar^+ ion sputtering and subsequent annealing. The primary contaminant was oxygen which continuously diffused from the bulk to the surface where it was removed by the sputtering until the bulk had been depleted to the point that annealing at 800K no longer resulted in the appearance of oxygen at the surface.

The friction measurements were all made between pairs of identical alloy samples whose surfaces were prepared using identical procedures. In order to allow the measurement of friction one of each pair was polished flat while the other was polished to have some curvature. Once aligned and then brought into contact these created a sphere-on-flat geometry. The friction experiment was performed by bringing the two into contact under a normal load of $F_N = 40$ mN and then sliding at $v_s = 20$ μm/s for a period of roughly 15 s. Both the normal force (load) and the shear force (friction) were measured simultaneously during sliding. The values of friction coefficients were calculated by dividing the shear forces by the nominal normal force measured at the point of beginning shearing. In this paper we report the values of the static friction coefficient or the friction force needed to initiate sliding. The details of the definition used in this work have been described earlier [30]. Since for the most part the friction force exhibited stick-slip behavior a meaningful value of the dynamic friction coefficient cannot be determined. In all cases the friction measurement was repeated 10-12 times using contact points between previously untouched parts of the surfaces. These then allow us to calculate an average and standard deviation for the friction coefficient.

The oxidation experiments were all conducted by exposing the surfaces to background pressures of 10^{-8} – 10^{-7} Torr of O_2 or H_2O at a temperature of 300K immediately followed by annealing to 600K. The uptake of oxygen by the surface was followed using AES. Since the two oxidation experiments were performed with different electron energy analyzers for AES there has been no attempt to compare the absolute magnitudes of the oxygen signals from the two samples.

RESULTS AND DISCUSSION

Oxidation of quasicrystal and approximant surfaces

As with any metal or alloy the surfaces of quasicrystalline materials become oxidized as a result of exposure to air. If they have some resistance to oxidation above and beyond that of other corrosion resistant metals then this becomes an important and potentially useful property. There have been prior studies of the oxidation of the AlPdMn quasicrystal surfaces which do provide insight into the chemistry of the surface oxidation process [10]. Exposure of a clean quasicrystal surface to oxygen results in the selective oxidation of the Al to form a thin passivating film of aluminum oxide. Once the surface is passivated the film will not increase in thickness during further exposure to O_2, however, it will thicken on exposure to H_2O vapor.

In the work described here we have studied the oxidation of the clean surface of the $Al_{48}Pd_{42}Mn_{10}$ approximant and compared that to our prior observations of the oxidation of the $Al_{70}Pd_{21}Mn_9$ quasicrystal surface. Oxidation of the alloy surfaces has been studied using AES to monitor the uptake of oxygen during exposure to both O_2 and to H_2O. Figure 1 shows the Auger spectra of the clean $Al_{48}Pd_{42}Mn_{10}$ approximant surface and the same surface after saturation with oxygen by exposure to O_2. The spectrum of the clean surface reveals the presence of all three expected elements with spectral features at the appropriate energies. In particular note that the Al peak position is 65 eV which is the position expected for metallic Al^0. Exposure of the surface to O_2 at 300K for a cumulative total of 319 L (1 L = 10^{-6} Torr•s) results in the adsorption of oxygen as revealed by the feature at 511 eV in the lower spectrum of figure 1. More importantly note that the Al feature has now shifted to 56 eV. The shift of the Al peak is consistent with the formation of an aluminum oxide phase. There is no evidence of any oxidation of the Pd or the Mn components of the approximant alloy. This oxidation behavior is qualitatively identical to that observed previously for the $Al_{70}Pd_{21}Mn_9$ quasicrystal [10,29].

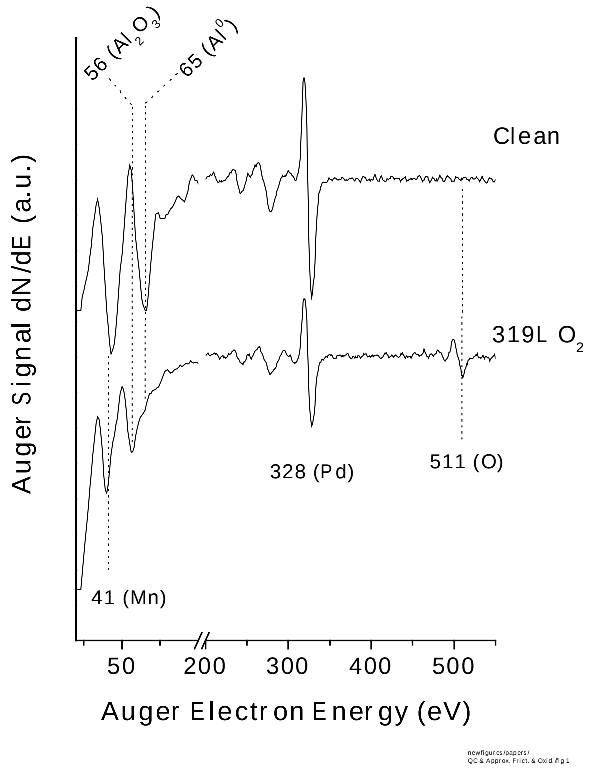

Figure 1. *Auger electron spectra of the clean $Al_{48}Pd_{42}Mn_{10}$ surface (upper) and the same surface saturated with oxygen (lower). The clean surface spectrum exhibits peaks from the three components of the alloy: Al at 65 eV, Pd at 328 eV, and Mn at 41 eV. Once oxidized a peak due to O appears at 511 eV. Also note that the Al peak shifts to 56 eV as a results of the formation of a film of aluminum oxide.*

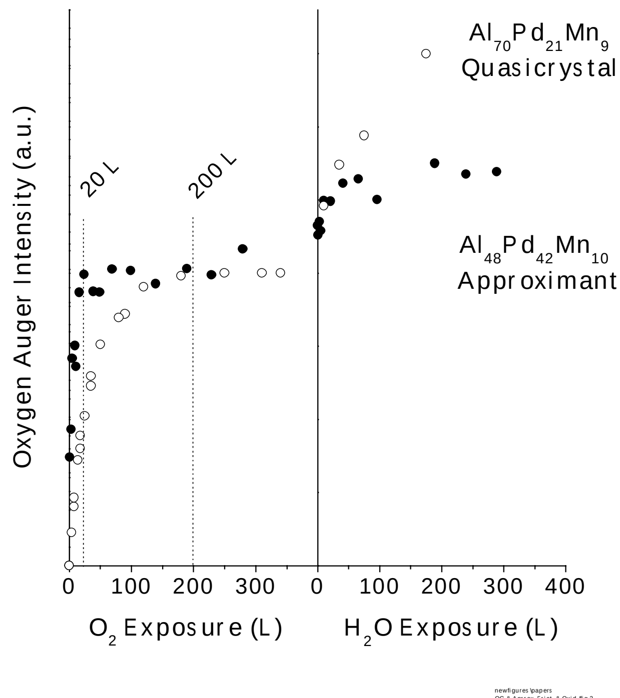

***Figure 2**. Oxygen uptake on the $Al_{70}Pd_{21}Mn_9$ quasicrystal (○) and the $Al_{48}Pd_{42}Mn_{10}$ approximant (●) surfaces as a function of exposure to oxygen followed by exposure to H_2O vapor. The exposure to O_2 results in the formation of a thin film of aluminum oxide which passivates the surface against further oxidation by O_2. Subsequent exposure to H_2O vapor results in a thickening of the oxide film followed by passivation at a higher film thickness. The rate of oxidation of the $Al_{48}Pd_{42}Mn_{10}$ approximant during initial exposure to O_2 is roughly ten times greater than the initial rate of oxidation of the $Al_{70}Pd_{21}Mn_9$ quasicrystal.*

The kinetics of the oxidation of the $Al_{48}Pd_{42}Mn_{10}$ approximant have been studied by monitoring the uptake of oxygen by the surface during sequential exposures to O_2. Figure 2 shows the amplitude of the O_{511} Auger peak (●) on the $Al_{48}Pd_{42}Mn_{10}$ approximant as a function of exposure to O_2. The oxygen coverage on the surface initially increases and then plateaus after an exposure of roughly 20 L. The implication is that the surface of the approximant becomes covered with a thin film of aluminum oxide that acts as a passivating film and prevents further oxidation. Subsequent exposure of the surface to H_2O vapor causes a slight increase in the oxide thickness but that too passivates the surface after an exposure of roughly 50 L. Figure 2 also shows the results of previous measurements of the oxidation of the $Al_{70}Pd_{21}Mn_9$ quasicrystal surface (○). Note that the amplitude of the Auger intensities for the oxygen peaks are not directly comparable since the measurements were performed with two different analyzers and so they have been scaled to give the same amplitude at saturation of the surface by exposure to O_2. Although the basic behavior of the oxidation of the quasicrystal and its approximant is the same it is important to note that the rate of oxidation of the quasicrystal is roughly 10 times lower than that of the approximant. Where it requires an exposure of only 20 L to saturate the surface of the approximant with oxygen it requires an exposure of roughly 200 L in the case of the quasicrystal. The other difference between the approximant and the quasicrystal is that, whereas the surface of the approximant is apparently passivated by exposure to H_2O vapor that of the quasicrystal is not, at least over the range of exposures used in these experiments. Nonetheless, the quasicrystal surface appears to exhibit some resistance to oxidation by O_2.

Both the $Al_{70}Pd_{21}Mn_9$ quasicrystal and the $Al_{48}Pd_{42}Mn_{10}$ approximant exhibit oxidation resistance in the sense that upon exposure to O_2 their surfaces become coated by a thin oxide film that resists further oxidation. This observation has been made in the past using the $Al_{70}Pd_{21}Mn_9$ quasicrystal surface and much higher exposures to oxidizing environments than have been used in this work [10]. Here we observe the same behavior with the $Al_{48}Pd_{42}Mn_{10}$ approximant. The additional and quite interesting observation is that the rate of oxidation of the quasicrystal is found to be roughly an order of magnitude lower than that of the approximant. Similar observations have been made with the icosahedral $Al_{63.5}Cu_{24}Fe_{12.5}$ quasicrystals and their approximants [22]. On the basis of stoichiometry one might expect the opposite behavior since the approximant has a lower bulk Al concentration than the quasicrystal and it is clear from the Auger spectra that it is the aluminum component of these materials that is most readily oxidized. The origin of the additional oxidation resistance of the quasicrystal over the approximant is intriguing but is not understood at this point.

Frictional properties of quasicrystal and approximant surfaces

The primary objective of this study has been to compare the frictional properties of well-defined surfaces of the $Al_{70}Pd_{21}Mn_9$ quasicrystal with those of its crystalline $Al_{48}Pd_{42}Mn_{10}$ approximant in order to address the question of whether or not quasicrystallinity itself plays an important role in determining their tribological properties. The ideal experiment in this regard would be a comparison of the friction between two materials of identical composition and mechanical properties whose only difference was that one had a crystalline rather than quasicrystalline structure. Of course, such materials do not exist and the only real way to address this problem is through a study of a set of materials which allow one to decouple the effects of various materials properties on friction and ultimately elucidate the role of quasicrystallinity.

This study represents only a first step along this path in that we compare the friction of quasicrystal and approximant alloy having similar but not identical compositions.

The role of UHV tribology in problems such as the one described in this work is to provide measurements of friction on well-defined surfaces where it is the properties clean surfaces rather than contaminant films that determine friction. In previous work we have measured friction coefficients of $\mu_s = 0.11 \pm 0.02$ between the surfaces of $Al_{70}Pd_{21}Mn_9$ quasicrystals in air and prior to any UHV cleaning [29]. These surfaces were, of course, coated with carbon and with an oxide film. Once cleaned and annealed to remove contaminants and produce a well-ordered surface the friction between two such surfaces rose to $\mu_s = 0.60 \pm 0.08$. Clearly, the low friction measured on the air-exposed surfaces is not intrinsic to the quasicrystals. Using the $Al_{48}Pd_{42}Mn_{10}$ approximant that had been exposed to air and were studied prior to any cleaning of the surfaces we have measured a friction coefficient of $\mu_s = 0.10 \pm 0.02$. Again these surfaces were highly contaminated and as will be seen this friction coefficient is not that of the truly clean surface of the $Al_{48}Pd_{42}Mn_{10}$ approximant.

Cleaning the surfaces of the $Al_{48}Pd_{42}Mn_{10}$ approximants requires extensive sputtering and annealing to remove oxide films and contamination. Once complete the surface gives an Auger spectrum such as that in figure 1 which reveals the absence of any oxygen and the presence of the Al in its metallic rather than oxidized state. Measurements of friction between the clean surfaces of the $Al_{48}Pd_{42}Mn_{10}$ approximants give a coefficient of friction of $\mu_s = 1.22 \pm 0.85$. Figure 3 shows three randomly chosen plots of shear force and normal force between the approximant surfaces during sliding. They reveal somewhat erratic sliding behavior. There are several points to make about the value of the friction coefficient between these clean surfaces. The first is that it is certainly higher than that observed under identical conditions using the $Al_{70}Pd_{21}Mn_9$ quasicrystals. The second point is that the friction between the $Al_{48}Pd_{42}Mn_{10}$ approximants is more erratic or exhibits greater stick-slip behavior than was observed between the $Al_{70}Pd_{21}Mn_9$ quasicrystals. This results in the much higher error bars on the measured value of the friction coefficients for the approximants. The most important observation, however, is that the friction between the $Al_{70}Pd_{21}Mn_9$ quasicrystals ($\mu_s = 0.60 \pm 0.08$) is lower than that between pairs of their $Al_{48}Pd_{42}Mn_{10}$ approximants.

Oxidation has already been shown to influence the friction between the surfaces of the quasicrystals [29]. It has been proposed that the low friction observed between quasicrystals in ambient air is due to delamination of the oxide film during shearing. We have measured the friction between the two $Al_{48}Pd_{42}Mn_{10}$ approximant surfaces as a function of oxidation of the surface during exposure to both O_2 and H_2O. The measured friction coefficients for the $Al_{48}Pd_{42}Mn_{10}$ approximant surfaces are shown in figure 4 as solid circles (●) and are compared to those previously measured for the $Al_{70}Pd_{21}Mn_9$ quasicrystals (○) in a similar experiment. As mentioned above the friction coefficient for the clean surfaces is $\mu_s = 1.22 \pm 0.85$. Oxidation of the surface with O_2 generates a thin passivating film of aluminum oxide that reduces the friction slightly to $\mu_s = 0.83 \pm 0.32$. Further oxidation does not have a significant effect on the friction which reaches $\mu_s = 0.71 \pm 0.20$. As in the case of the $Al_{70}Pd_{21}Mn_9$ quasicrystals, oxidation of the surfaces of the $Al_{48}Pd_{42}Mn_{10}$ approximants does lower the friction coefficient. At all levels of oxidation, however, the friction coefficient of the $Al_{48}Pd_{42}Mn_{10}$ approximants is higher than that of the $Al_{70}Pd_{21}Mn_9$ quasicrystals. At this point the origin of this difference is not clear. While it is certainly tempting to attribute it to the fact that one sample is quasicrystalline while the other is not it must be kept in mind that there are other differences between the two samples that result from their difference in stoichiometry.

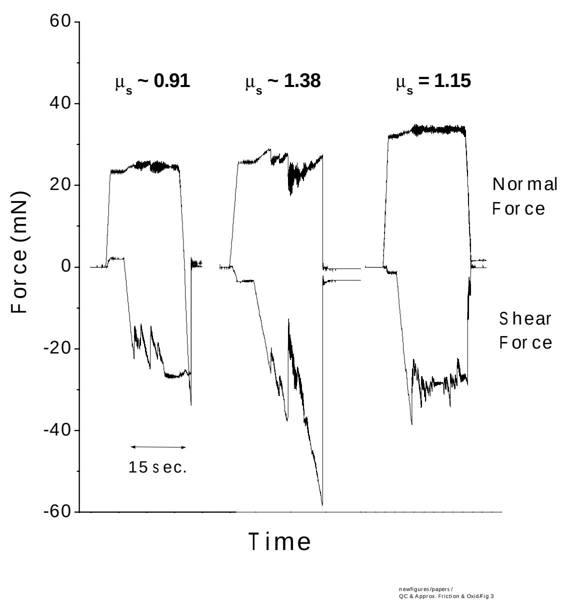

Figure 3. *Three friction measurements made between clean $Al_{48}Pd_{42}Mn_{10}$ approximant surfaces. The upper trace is a plot of the normal force, while the lower trace is a plot of the shear force. Friction behavior between the approximants was found to be more erratic than for the quasicrystals. Adhesion can be seen (left trace) as indicated by a negative normal force upon surface separation. Stick-slip friction was also observed for the approximants (middle trace).*

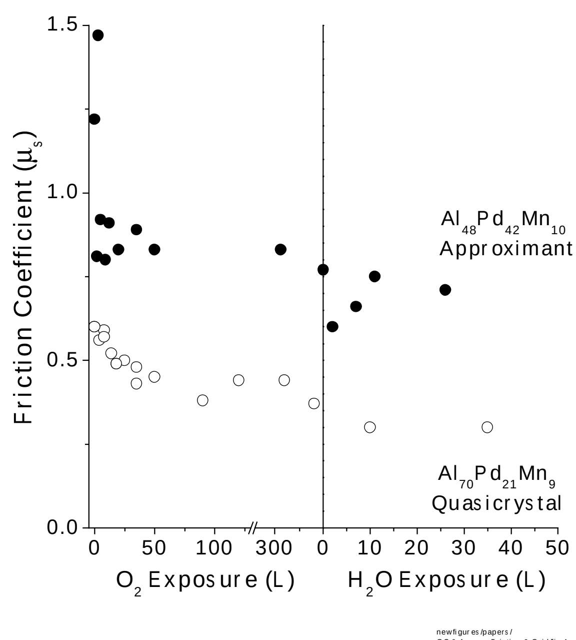

Figure 4*. Static friction coefficients measured between the surfaces of pairs of $Al_{48}Pd_{42}Mn_{10}$ approximants (●) and between pairs of $Al_{70}Pd_{21}Mn_{9}$ quasicrystals (○) as function of surface oxidation. For both samples the friction coefficients are reduced by oxidation. At all levels of oxidation the friction between the $Al_{70}Pd_{21}Mn_{9}$ quasicrystal surfaces is lower than that measured between the surfaces of the $Al_{48}Pd_{42}Mn_{10}$ approximant. The conditions used for the friction measurements were $F_N = 40$ mN, $v = 20$ μm/s, $T = 300K$.*

CONCLUSIONS

The experiments reported in this paper illustrate the need for and the value of UHV measurements in determining the properties of quasicrystalline surfaces. We have studied the oxidation and the friction of an $Al_{70}Pd_{21}Mn_9$ quasicrystal and its $Al_{48}Pd_{42}Mn_{10}$ approximant under conditions where their surfaces are free of contaminants that would otherwise mask the intrinsic surface properties of these materials. While both materials exhibit oxidation resistance in the sense that they become passivated with thin oxide films that prevent further oxidation, the rate of oxidation of the $Al_{70}Pd_{21}Mn_9$ quasicrystal surface is roughly 10 times lower than that of the $Al_{48}Pd_{42}Mn_{10}$ approximant. Comparison of the static friction coefficients between pairs of the quasicrystals and pairs of the approximants reveals that the friction of the quasicrystal is roughly ½ of that between the approximant surfaces. The origins of these differences cannot be determined categorically from this set of measurements, however, they do reveal that there are substantial differences between two alloys of similar composition but differing in the nature of their bulk structures in that one has a periodic crystalline structure while the other is quasicrystalline.

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