SURFACE MODIFICATION BY MAGION DEPOSITION - A FUTURISTIC PROCESS THAT IS SHAPING THE PRESENT THROUGH ADVANCES IN SURFACE TECHNOLOGY

by

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INTRODUCTION

Magion deposition is a new process by which virtually any material may be tightly bonded to nearly any other material for any reason.[1] This process uniquely utilizes ionization phenomena to impart high energy to the atoms of plating material whereby bonding is assured by penetration of these high energy depositant ions into the outer atomic surface of the object to be plated, which is referred to as the "substrate". In the actual working of the process, which is performed in a vacuum system, the plating material is vaporized yielding a continuous flux of atomic sized vapor atoms. These atoms become ionized by a twofold mechanism, collision with electrons spiraling in a magnetic field as well as by resonance with photons in a radio frequency (rf) transmission field. The rf field also has the beneficial effect of building a negative charge on non conductors present in the field. This causes both conducting and non-conducting substrates to become attractive targets for the high energy, accelerating ions. This allows for the process to treat both metals and elastomers as equals. The three dimensional properties of the process are unusually good. One of the first arenas for success was the plating of electronic components such as semiconductors and printed circuits where through hole or side step coverage can be of paramount importance.[2] This thin film process eliminates over a dozen wet chemistry plating steps in device manufacture resulting in a superior product at a much lower cost. Perhaps the biggest bonus of all is the elimination of wet chemistry waste products that must be disposed of at a very high cost. Contemporaneous with the advancement of the Magion process has been metallurgies appropriate for the specific problems at hand. The development of specific metallurgies, most not capable of deposition in the right alloy form by any process, was essentially necessary before the Magion process could be employed to yield a finished product. One gauge of success to date is to note the current licensees and their field of usage. Many of these will be discussed at length in the body of this paper. An important consideration will be protection of the environment that this technology offers. The best way of dealing with pollution is to remove the source of its generation. New developments in elastomeric seal technology aid in pollution control.

COMPARISON WITH PREVIOUS VACUUM PROCESSES

Physical Vapor Deposition

The first vacuum process to be utilized for industrial coatings was that of simple evaporation. Early systems were quite simple and the process involved merely placing a substrate in the line of sight from an evaporant source (usually a tungsten filament) so that it could receive and condense metal vapors from the hot evaporant source. This process is still widely used and, as the systems themselves have become increasingly sophisticated, the process has come to be known by the more elegant name of Physical Vapor Deposition (PVD). The line of sight characteristic became more and more of a problem as the field of integrated circuitry developed with its attendant requirements for uniform deposition in holes and along walls. Devices such as planetary systems and nutating holders, (nodding, rocking or oscillation) all very expensive, have been developed to improve the situation but these are not entirely effective. It should be emphasized that the material transport mechanism in PVD is merely that of a change of state from solid to vapor, then following a line of sight path to condense back to solid on the first cold surface that the evaporant stream hits. There is absolutely no means for acceleration of these evaporant atoms to result in a high energy impact on the targeted surface. This is essential if the binding adhesion is to be any better than that resulting from simple condensation. Figure 1 shows a schematic of a PVD system. These PVD systems are in use in the world today and have a billion dollar impact on the gross national product. This technique is still the most cost effective for making large numbers of inexpensive objects bright and pretty. This industry is referred to in the trade as "junk coating", the term junk referring to the objects being coated, not the systems themselves. Quite the opposite, the large evaporative coating systems now in use have had several generations to evolve into the reliable, cost effective systems. When one compares both the price and reliability of these systems with some of the new, ultra-sophisticated process/systems in state of the art electronics applications today, the results are appalling.

Sputtering process

The next vacuum deposition to attract industry attention was the "sputtering" process. This system is illustrated in Figure 2. The recognition of the "glow discharge" goes back to the 1850's. Sir William Crooke, in 1867, further described the physical characteristics of the glow in the vicinity of the cathode, giving his name to this thin, dark region between the visible plasma and the negative electrode that we now call the "Crooke's Dark Space". For the next 100 years there were only about 100 papers written on what ultimately became known as the "sputtering" process, with the majority of these being on ways to prevent it. Curiously enough, it was a fairly widely recognized failure mechanism in early experiments with vacuum devices. The process began to be seriously considered as a means for material deposition in the mid 1950's. The first usage was in dc systems which utilized negative biased cathodes made of evaporant material with the substrate mounted on grounded work tables. The later development of rf sputtering offered a means to deposit dielectric materials and was the first to enjoy broad industrial usage. This was the type of sputtering process that was used to deposit a thin film of platinum for protecting the cutting edges of razor blades. Most modern sputtering systems are of the planar magnetron configuration which is a dc system with deposition rate enhanced by a magnetic field associated with the cathode.

One can see by inspection of the schematic of the sputtering system that it is a secondary process. The application of the accelerating voltage is not to the atom to be deposited but rather to the argon ion that bombards the cathode to liberate a neutral atom of cathode material. Note that the depositant atom must be neutral else it could never escape the attraction of the cathode field. The atom further undergoes decelerating collisions on the way to the substrate. Even so, its arrival energy is somewhat greater than that of PVD, and the collisions do partially alleviate the line of sight problem. Film adhesion still leaves something to be desired.

Ion Plating

A significant breakthrough in deposition technology occurred in the mid-1960's when researchers at the Sandia Laboratories successfully combined some of the better features of PVD and sputtering into a new process which exhibited some unusual features. The process was called Ion Plating and is illustrated by Figure 3. In this system, evaporant atoms pass through a plasma of argon gas wherein ionizing collisions with argon atoms occur. Up to 5% of the atoms are ionized by this mechanism and are immediately accelerated toward the dc biased substrate. The remaining 95% proceed as in PVD. The intense argon bombardment does contribute to film adhesion by a "hammering in" effect. As it turned out, there are as high as 30 atomic percent of argon atoms entrapped as the film builds. Since the full cathode voltage drop can be applied to ions enroute to the substrate, interfacial ion burial can result in values of adhesion over 1,000 times greater than that of PVD or sputtering. The three dimensional quality of the film deposition, enhanced as it is by the low mean free path argon collisions, tends to be excellent. This technique did not meet with widespread industrial acceptance for several reasons. It is not readily adaptable to non-conducting substrates. The entrapped argon results in poor repeatability for such important items as film sheet resistance and specularity. Perhaps the most serious impediment was the side effect that occurred within the system of polyatomic particles of evaporant aggregating in the argon cloud and depositing as a sooty layer over all of the system components. This curse to good vacuum operations resulted in a continual need for system cleanup. Clearly something had to be done to circumvent these difficulties if a general case, high throughput and cost effective process were to be developed.

Figure 4 shows a schematic of the "Magion" deposition system which represents the latest in the state of the art in vacuum technology.[3] This system circumvents the above listed problems by the utilization of an entirely different ionization mechanism, actually two independent mechanisms, that result in the establishing a confined plasma of only the evaporant ions. First is the application of rf energy to the substrate which becomes a radiating antenna. This subjects incident atoms of evaporant to photon bombardment in a resonant rf cavity which is a classical means for ionization. Second, in order to increase both the intensity and control the location of the plasma, a magnetic field is established at the substrate. Thermionic electrons from the heated source as well as Townsend avalanche electrons from the plasma itself are captured in spiraling paths by the magnetic field around the substrate, offering a high ionization cross section to atoms on the way to the substrate. These two mechanisms combine to put the maximum plasma density immediately around the substrate, which is the point in the system where it is most desired. The plasma of Figure 3 is primarily that of argon gas; whereas that of Figure 4 is the pure composition of the depositant without impurity of argon or any other ionizing gas. This intimate proximity of plasma to the substrate is an important feature of the Magion process resulting in many benefits. The sequence of events is simple in that the source feeds atoms of evaporant to the plasma, wherein they become ionized and are immediately fed on to the substrate at a very high velocity by the electrostatic attraction across the dark space. This provides for excellent film adhesion as well as good three dimensional uniformity.

Figure 5 is illustrative of the adhesion attainable by the Magion process. Here the adhesion of the gold to the glass was sufficient for the pull test stud to actually pull out a piece of glass from the substrate. Table 1 gives some indication of the magnitude of adhesion values that can be obtained over a wide variety of plating and substrate materials. These values were obtained by a Sebastian pull tester which applies a direct, measured force per unit area to accurately determine a numerical value for the true film adhesive strength. The adhesion inherent in the process is further illustrated by the SEM (Scanning Electron Microscope) photo of Figure 6. In this case, a film of aluminum, one micron thick, was Magion plated onto a nylon pull test specimen. The nylon specimen was then pulled apart in a tester and the SEM photo was made of the necked down region. Note that the aluminum actually parted and flowed along as small islets still tightly adhered to the base nylon. No peeling or flaking of the coating was observed.

A capability that is inherent in every Magion system may be inferred from the schematic of Figure 4. If argon were to be admitted to the system in the 10- 20 micron pressure range and the rf power supply were energized, then the rf sputtering process would commence. This is actually done in each and every cycle of a Magion plater and is referred to as "backsputtering". The term derives from the fact that material is actually being removed from the substrate. This material is mostly adsorbed water vapor and other surface residuals. This is a wonderful and convenient time to do a last level of cleaning by literally shot peening with atomic sized particles. This alone is one of the reasons for increased adhesion of the deposited film when compared to other vacuum processes. It is important to note that surface preparation is of utmost importance in vacuum processing. Organic surface contaminates tend to volatilize and are fragmented by the ionization and may even be deposited as a film impurity. This consideration lends credence to the need for backsputtering in any form of vacuum deposition.

The need for three dimensional coverage manifests itself under a variety of terms in many industries. It may be side-step coverage in integrated circuit manufacture, through hole plating in printed circuit manufacture or thread profiles in the case of metal lubricant films to prevent thread galling. Although expensive and complex presentation systems are used in microelectronics systems to provide step coverage at the micron level, the results often look like those of Figure 7. Note the poor coverage along a coincident side step. This coating was done in a very elaborate PVD system (an electron beam gun with a full rotation substrate holder) and yet the discontinuities can be seen. In the case of Figure 8, the original wafer of Figure 7 was cleaned and re-coated in a Magion deposition system. The mounting was simple and there was no elaborate substrate motion to enhance coverage. The comparative results may easily be seen in Figure 8. Note the smooth and uniform coverage of the side step. During deposition, the substrate was placed on a flat table, inclined 30 degrees from the source, and there was no rotation.

PROGRESS OF DEVELOPMENTS

Three dimensional coverage is vital to surface modification applications in quite a number of industries. Perhaps the best illustrations of this unusual property of the Magion process can be seen in more photos from the electronics industry. Figure 9 shows complete coverage of a "blind via" in a high density printed wiring board. The crossectional view shows the original outer cladding with a laser drilled hole that is 7 mils diameter at the entrance to the cusped hole. The one micron thick Magion plated copper covered the hole completely and was then placed up to 1 mil thickness by conventional electroplating. Applications such as this are proceeding under license at Texas Instruments in the US and at Lintek in Australia. Of these, the Lintek operation is by far the most noteworthy due to its capture of a significant portion of the printed circuit business in a large area of the Pacific Rim. The economic impact of the Magion process on this segment of industry was first recognized by these Australians and stems from two basic attributes of the process. The first of these is the potential for finer line resolution and superior adhesion over conventional wet chemistry board manufacturing. The second, and of greater manufacturing cost effect, is the elimination of pollution and waste handling problems that are currently associated with the wet chemistry processes. The Magion approach has eliminated 15 steps of wet chemistry with all their attendant costs. The net result is the elimination of over 50% of the shop costs of board manufacture, while producing a superior product.

Early in the development of applications for Magion plating, it became obvious that it would be short sighted to restrict it to a particular industry. As mechanical application out paced electronic usage it became evident that "Surface Technology" and Surface Modification" were more descriptive term for the Magion process. The field of "Tribology" (the study of lubrication, friction and wear) began to show fruitful applications such as those of Figures 10 and 11. The aircraft splines of Figure 10 presented a problem to operators that resulted from "Fretting" type failure. This has been a difficult problem in a lot of applications because it is not well understood. The solution proved to be both simple and inexpensive. This increased service life ten fold. It consisted of the application of a phased matrix of hard-to-soft metals, in this case chromium and gold, that allow for dry metal lubrication in much the same way that an oil impregnated bearing works. The Cr/Au system is further resistant to many of the harmful reagents that occur in the production of sour oil wells. This problem can be easily solved with high alloy metallurgy but is costly. There is a tendency for galling when these metals are in rubbing contact with each other, and conventional lubrication is ineffective or even impossible. Thus, the high alloy sub-surface safety valve balls of Figure 11 were a logical, and instantly successful, application of the technology. This has been done for this company's products without field failure for over 10 years.

The next logical extension of the technology was in the solution of the problems associated with the galling of high alloy oilfield threads. This was first reported to the industry in 1883.[4] The state of the art in those days in regard to this problem was deplorable. Primarily in the interest of getting high alloy tubular goods on the market, the manufacturers had an array of about 15 different methods for treating threads to prevent galling. All methods had one thing in common. Chemistry put them on and chemistry, found in sour flow streams, could easily take them off. High percentage of galled threads on rework was a certainty. The use of copper plating as an integral part of high alloy flow systems is questionable. Copper's affinity for attack by reagents present in sour flow streams discourages the use of high alloy tubulars. Copper does, indeed provide for a good initial run-in which enables the completion rig to move off in good shape leaving the problem to the next workover. The problem of high alloy tubular galling has been solve by the Magion chrome-gold system. This application has been licensed to Baker Hughes who offer it worldwide under the trade name of "Bakertron". It is interesting to note that many of their customers have been quite successful in running high alloy tubular strings using only 10 weight motor oil as pipe dope. This has the further advantage of getting rid of formation damage problems associated with pipe dope.

Two difficult problems, which were considered to be merely a cost of doing business and for which no solution was being sought have been solved. The first of these was a very severe bearing problem associated with a rotating liner hanger then manufactured by Lindsey Completions, now Smith Industries. The problem resulted from the need to rotate the casing string under loads that often exceed 100,000 pounds. The space available in the design of the tool to accommodate conventional bearing elements and races was limited. The solution was a four element dry metal lubricant system applied directly to the inner surface of a metal ring that operates in full metal contact with a mating rotational member. This method has been in successful usage for over 5 years. The economic comparison of the Magion plated ring (a $100 cost and 48 hour delivery) with the nearest competitor from the bearing industry (Astroseal cost $1,500 with 14 week delivery) only serves to illustrate the degree of absurdity with which obsolescent methods hold on for dear life against the encroachment of new and innovative technology. The second of these unusually severe problems was that of the Merla (now Halliburton) all metal casing patch. The design of this tool allows for wireline setting. As the powder squib fires, a setting force of near 100,000 pounds causes a tapered mandrel to stroke inside a mating tapered metal sleeve. The metal shell is actually swaged outward to contact the ID of the tubular being patched by forming a hermetic seal. The major problem holding up the development of this tool was the tendency of the contacting metals to gall during the swaging stroke. This imparts a much greater contact stress than normally associated with galling since it actually takes the metal past the yield point. The stainless steel parts increased the galling problem. The same four element dry quartenary metal lubricant film that was used in the liner hanger bearing was applied to this problem with an instant success. The above has been in successful field service for several years.

ONGOING DEVELOPMENTS

One of the logical outgrowths of advancement of technology is the improved skill level of the practitioners in asking questions regarding the nature of failures. More often the solution to a problem stems from the ability of the investigator to ask the right questions. The Magion process offers a virtually unlimited choice of materials which can be applied to a particular surface related problem. A correct understanding of the mechanism of failure may well be the key to the recommendation of a material film. A perfect illustration of this is found in the early work at applying Magion metal lubricant films to the solution of elastomer failures. A basic and fundamental question revolves around the nature of elastomers.

In the process of degrading in seal bores, elastomers form hard residuals that are extremely difficult to clean up during servicing. Why does it require silicon carbide paper to clean up after a stuck seal that has usually torn up during removal? The answer to this lies in the failure of the cross linking bonds that the rubber chemists employ during molding to cause the elastomer to retain its shape and not behave as a liquid. In this failure, a free carbon is yielded that subsequently finds a reactive metal in the seal bore, and the result is the formation of a metal carbide residue that is difficult to remove. Once this was recognized, it became logical to treat the failure in an unusual way with Magion deposited films. The initial deposition logically requires a ready carbide former at the boundary layer with the elastomer surface. This can be from a variety of materials such as titanium, chromium, zirconium or even in some cases pure iron. If carbon migrates from the body of the seal to the seal bore, then a barrier film such as nickel is further logical. Nickel is an effective barrier to carbon due to mutual insolubility and the fact that nickel carbide does not exist in the solid state. The method becomes one of corralling the carbon within the plated metal film and out of contact with the seal bore. To further enhance the operation of the seal, it is both possible and advisable to apply a metal lubricant film such as bronze to the outer surface. This enhances removal at end of service for static or in continuous operation for dynamic seals. Applications on oil well stuffing box rubbers and gas lift valve chevrons are examples of the above.[5]

The motivation to improve the state of the art in stuffing box packing was initially to provide the user with an improved approach to reducing the emissions from stuffing boxes along with their attendant liabilities. This was particularly well presented in the paper by Leslie Savage of the Texas Railroad Commission.[6] Work has proceeded well in addressing the problem of seal sticking in the bores of side pocket mandrels. Such failures of elastomeric seals and the attendant damage to the mandrel pockets has long been ignored in the gas lift industry, by both suppliers and users. This work was reported by White.[7] One of the most difficult aspects of the early stages of the packing project was the evaluation of various candidate rubber compounds for the Magion coating. It was determined experimentally that not all compounds are suitable for the physics of the Magion process. Not only did the coating itself have to be optimized, but the base elastomer as well. Success was best achieved with a peroxide coated buna-n compound. The paper by Angelo cites some noteworthy successes from lab tests on Magion coated packing.[8] It should be noted that these were from a very different compounds than the one that previous field tests had shown to be the best. Unfortunately, there was inconsistency in the effort of Angelo.

By far the most graphic results are from efforts to reduce the stem leaks from motor valves. Seal failure in motor valves is largely the result of scale deposits on the stem "filing" on the seals during stroking with cutting as the inevitable result. If the scale has very little adhesion, it will merely be wiped off by the seal and the results shown in Figure 12 and 13 are now becoming routine the world over. This cost effective process is now being routinely applied to gas-lift valves. The result is that the frequency of valve replacement has been lessened by a factor of three. When planning field operations, the impact of such operating cost reductions should be throughly analyzed and factored into overall well cost projections. Economic savings can be enormous. If lease operators neglect to reduce failures, it only perpetuate the suppliers dream of a highly profitable and dependable aftermarket.

The reader is urged to pay particular attention to Figures 12, 13, 14 and 15. These illustrate the interrelation developed between the properties of surface lubricity and hydrophobicity in addressing an even broader range of failures. This was made possible by the development of hydrophobic surfaces to resist the formation of scale. Hydrophobia literally means fear of water. The problem of scale deposition comes about from water borne solids dropping out of solution to deposit on a surface. Most oil wells produce an oil/water mixture. It was a logical choice to experimentally select an optimized alloy that would exhibit a natural tendency to keep itself oil wet by continuous replenishment with oil from the flow stream while simultaneously rejecting the water component. The results to date, as reported by ARCO, have been most encouraging.[9]

CONCLUSIONS

The bright and broadening future of surface technology in general and the Magion process in particular seems now to be assured. The best indication of this may well be in the nature of present developments that have showed successes. These would the power generation industry, the circuits industry with more recent attention being paid to flat panel displays, aircraft industry fasteners and components and bearings for a variety of applications including X-Ray machines. It is hoped that the current license to Texas Tech for research using the Magion process will bear fruits in many areas heretofore not even considered.

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Table 1 - Adherence Tests

Substrate Material	Deposited Material	Thickness (KA)	Adherence (lbs./sq.in.)
7033GL (glass-reinforced nylon)	Aluminum	3	7720
7033GL (Dupont)	Nickel	3	4620
11C40 (minlou) (Dupont)	Aluminum	50	4690
Rynite (Dupont)	Aluminum	3	5290
Teflon (Dupont)	Aluminum	50	8880
Ryton (Phillips 66)	Nickel	3	4920
Torlon (Amoco)	Ni/Cu	10	5130
Polysulfone (Union Carb.)	GTH	3	10400
Glass	Ni/Cu	7	8850
Steel	Titanium	3	>10500

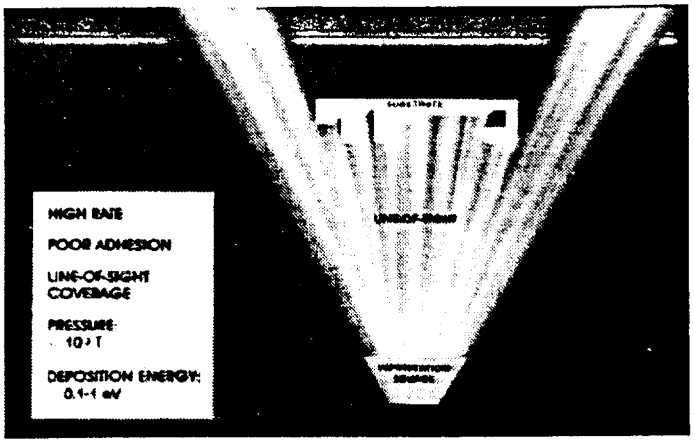


Figure 1 - Schematic of Physical Vapor Deposition (PVD) process

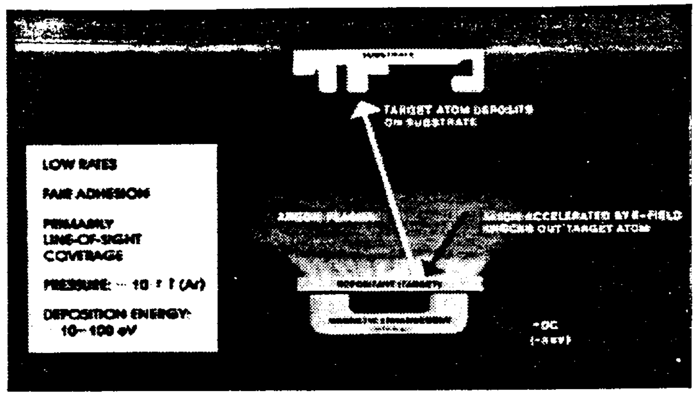


Figure 2 - Schematic of Sputtering process

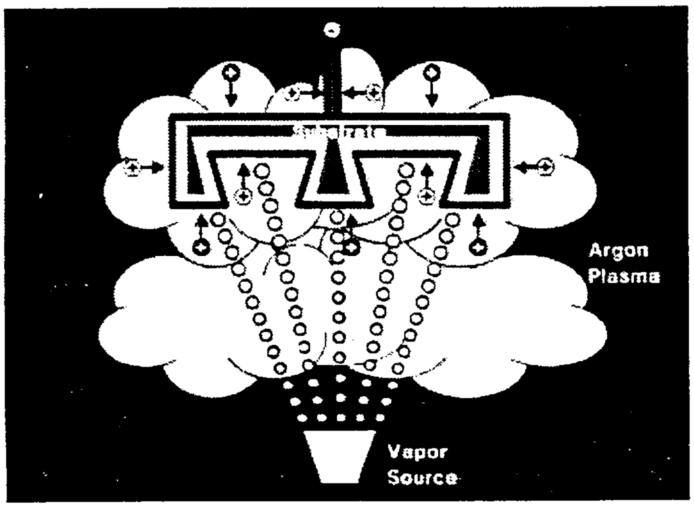


Figure 3 - Classical Ion Plating process schematic

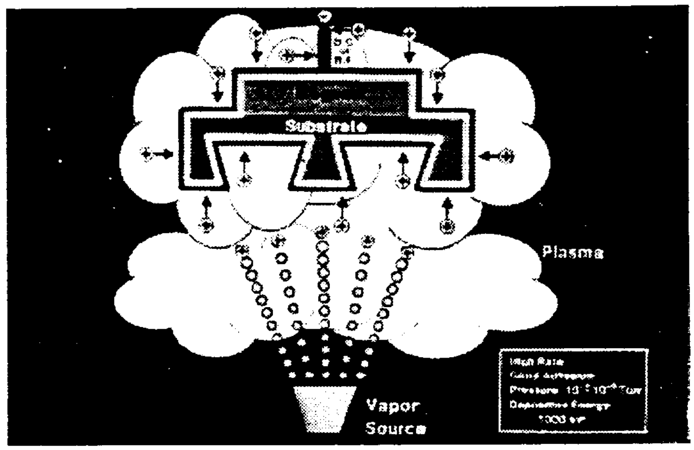


Figure 4 - Magion deposition process schematic

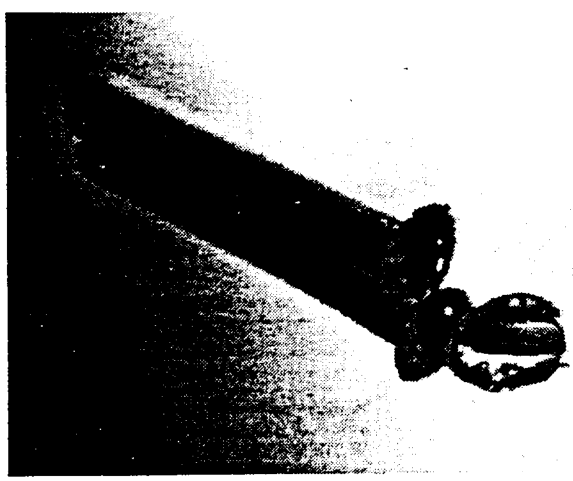


Figure 5 - Glass slide with Magion plated Cr/Au film after pull test. The glass pulled out in tension at 3500 psi.

Figure 6 - The scanning electron microscope looks at the failed region of a nylon pull test specimen that had been pulled apart in a tester. Note the adhesion of the 1 micron thich Magion plated film of aluminum that has parted in tension but not peeled.

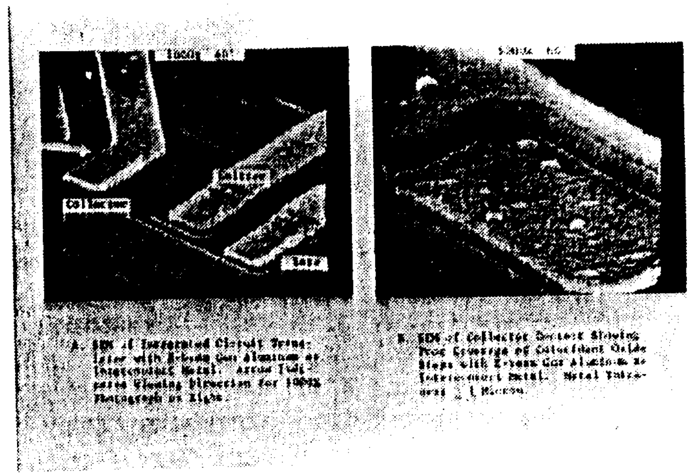


Figure 7 - An integrated circuit (7000 x size) that has been metallized with a micron of PVD aluminum. Note the poor coverage along a coincedent side step. The metallization was done with an electron beam gun with full rotation substrate holder.

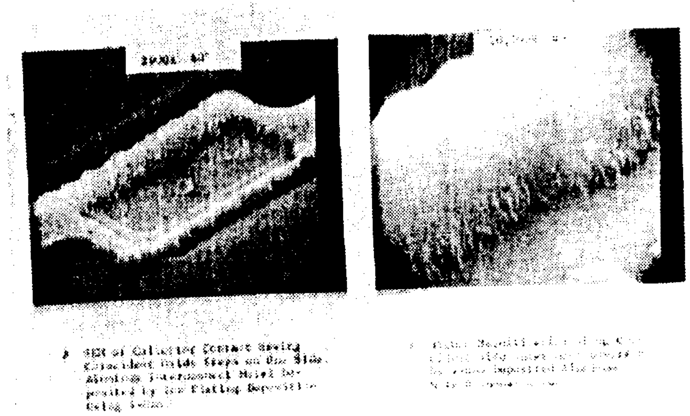


Figure 8 - The very same ic of figure 7 with the PVD aluminum removed and replaced by identical thickness of Magion deposited aluminum

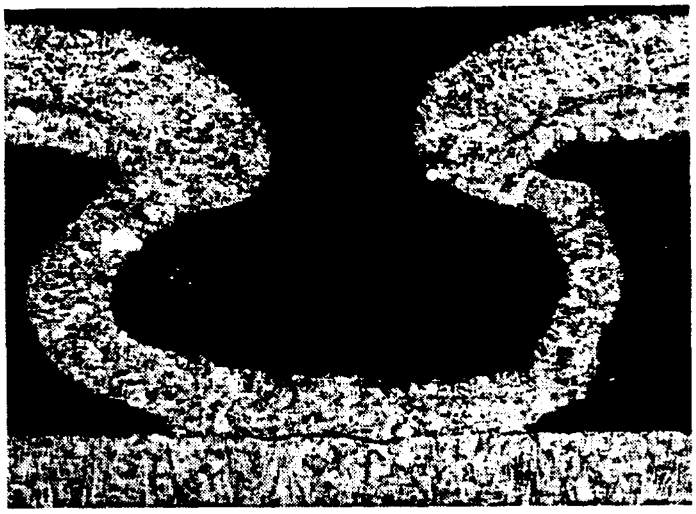


Figure 9 - Cross sectional micrograph of a Magion plated "blind" via in a high density printed wiring board

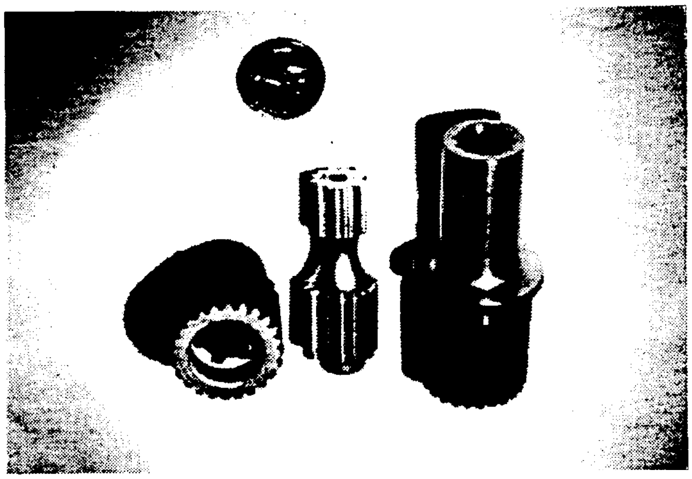


Figure 10 - Aircraft splines that have been Magion plated with chrome/gold film to prevent fretting failure

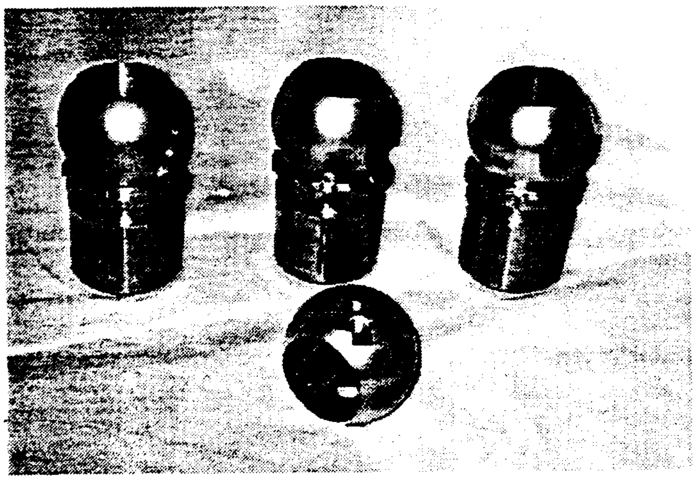
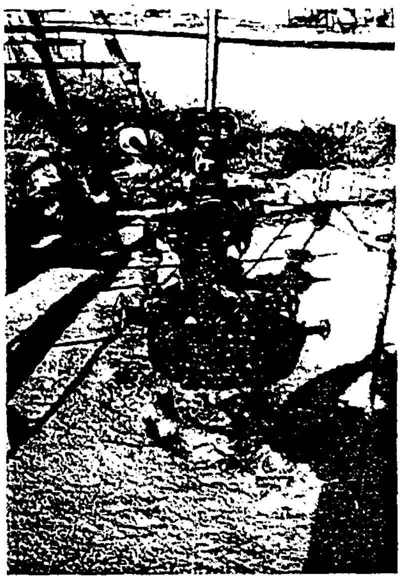


Figure 11 - Sub Surface Safety Valve ball elements that have been Magion plated with chrome/gold



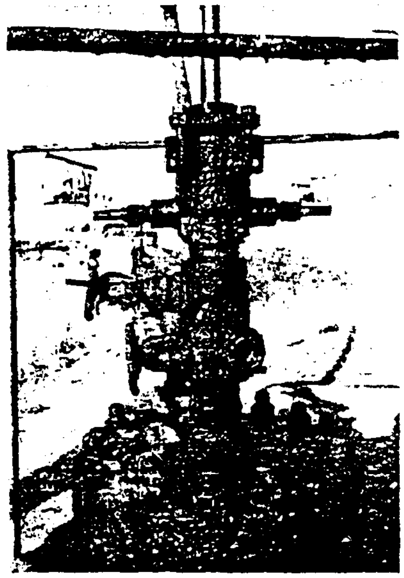
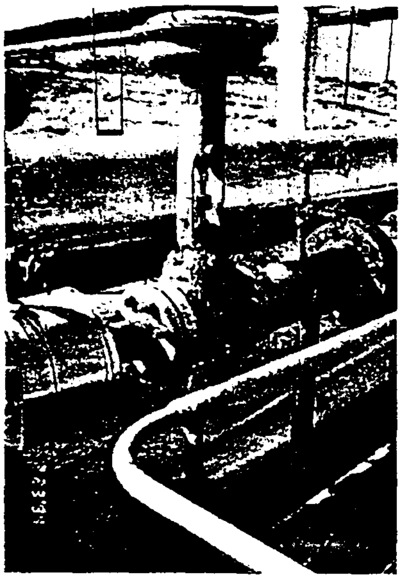


Figure 12 - Service performance comparisons of Magion plated packings versus conventional packings.

Figure 12a - The rubber cones in the box have been Magion plated with "White Gold" Metal Lubricant films for emission control through higher installation compression and longer service life.

Figure 12b - Conventional rubber cone packing show gross evidence of fugitive emission in the exact same service as those in Figure 12a. They are in use in steamflood pumping heavy oil.



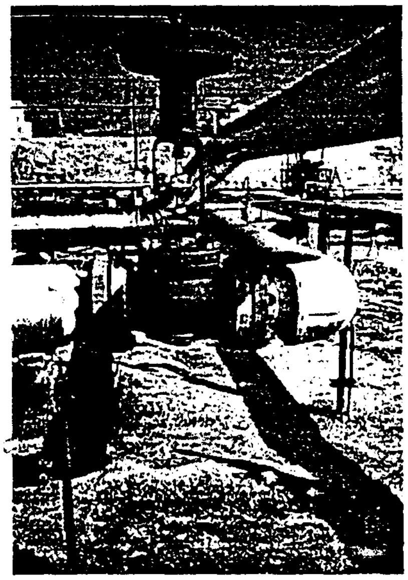


Figure 13 - Direct field comparison of motor valves with and without "White Gold"™ Hydrophobic metal lubricant film

Figure 13a - Motor valve with conventional stem after the same time in service as the one in Figure 13b. As the scale builds up on the downstroke side, it acts like a file to cut the seals causing the uncontrolled release of fugitive emissions. Contrast this valve with the Magion plated valve of Figure 13b. The conditions of service for both were steam with water treatment residue at 1,000 psi and 600 degrees Fahrenheit.

Figure 13b - The stem in this motor valve has been Magion plated with "White Gold"™ Hydrophobic metal lubricant film to resist the formation of mineral scale. This valve has been in service for over a year in conditions exactly equal to those of the valve of Figure 13a.

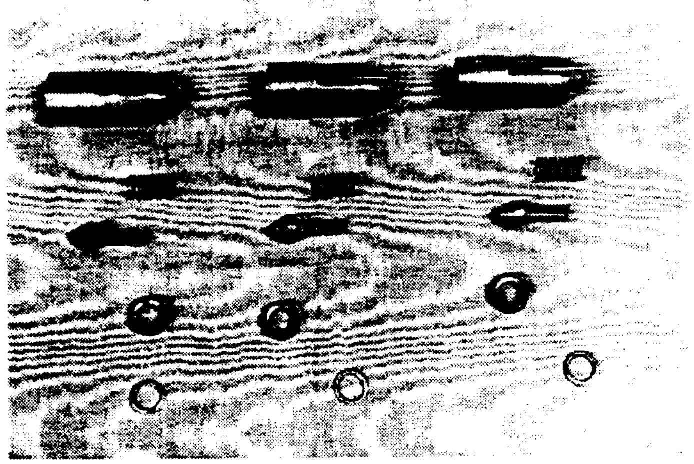
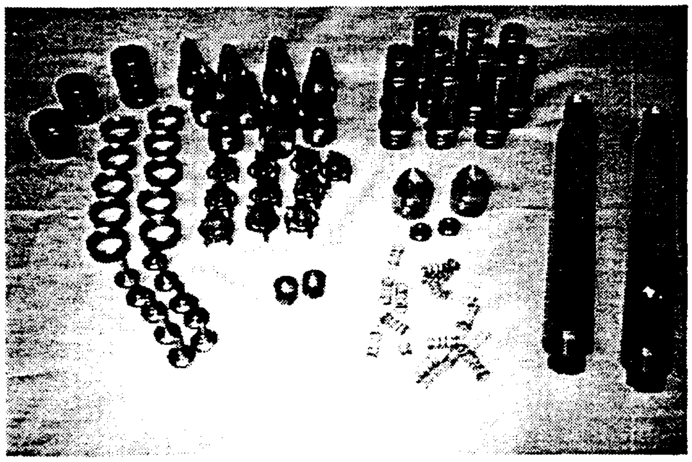


Figure 14 - These sub-parts for the Otis Merla Systems wire line retrievable chemical injector valve were Magion coated with a scale resistant metallurgical system. After assembly, the valves were run by ARCO at Farmington, New Mexico in wells that were notorious for scale production. After 120 days service in a well where scale plugging was normal after 90 days service, the valve was pulled for examination and was found to be scale free and was subsequenly rerun with no further treatment.



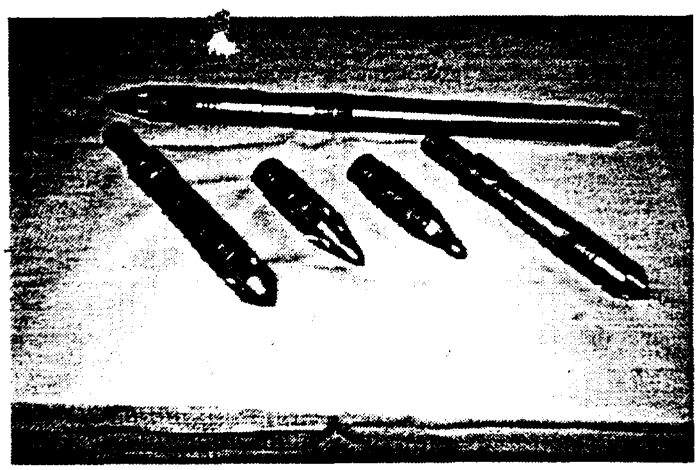


Figure 15 - Halliburton (Merla) gas lift valves with "White Gold"™ Hydrophobic Metal Lubricant Films for operation in oil wells in Prudhoe Bay. This treatment is now standard for both valves and chevrons in stalled in Prudhoe Bay.