Sputter-Seeded Activation of Electroless Nickel on Composite Alloys

Leonard Nanis[*]
LN3 Electrochemical Engineering
San Jose, California USA

A novel sputtered seed layer method of activation for plating electroless nickel has been applied to difficult-to-activate particle reinforced aluminum, a new breed of high-strength lightweight alloys. Basically, in one single step, sputtering (PVD) deposits a thin film seed layer which covers all phases and gives the substrate surface a new face. The seed layer for Ni-P plating is sputtered from a target of 88% nickel-12% phosphorus alloy. When immersed in the electroless nickel bath, the new face actively and uniformly "jump-starts" adherent conformal growth of fresh layers of Ni-P from the bath chemistry. From the viewpoint of the electroless reaction, the sputtered atomic layers of Ni-P appear as already "deposited" material, available to nucleate new Ni-P layers autocatalytically. Sputter-seeded activation completely replaces all steps of conventional double zincate pre-treatment of aluminum alloys and is particularly effective with composites. Composites are strengthened by the inclusion of micron-sized hard particles, such as silicon carbide (10 to 40 vol%) mixed throughout a matrix of aluminum. Since the silicon carbide phase does not participate in the zincate reaction, the surface of the composite alloy is incompletely activated by conventional zincate pre-treatment and the Ni-P deposit contains numerous deep pits that begin at SiC particles. By contrast, both matrix and particle phases of a composite are activated by the sputter-seeded layer and Ni-P grows smoothly, without pits and essentially without nodules. Sputter-seeded activation permits Ni-P to be plated on non-metals, such as glass, as well as on metals which do not respond to zincating. To insure adhesion to the substrate, a thin binder layer of chromium may first be sputtered onto the substrate before the seed layer. Both binder and seed layers are typically 200 to 500 Å thick, achieved rapidly by sputtering. The seed layer remains active for several days, thus permitting a delay time between sputtering of the seed layer and Ni-P plating.

Keywords: *Electroless nickel, sputter-seeded activation, plating on aluminum*

Introduction

A novel sputtered seed layer method of activation of aluminum for plating electroless nickel has been reported.[1,2] The sputter seed-plate method has now been successfully applied to a new breed of aluminum alloys strengthened by the inclusion of hard particles, a type of alloy also known as metal matrix composites (MMC).

Basically, in one single step, sputtered seed layer activation puts a new face on a substrate material (Sputtering is also known as physical vapor deposition or PVD.). Sputtered nickel-phosphorus alloys are effective seed materials[3] for electroless nickel plating. When immersed in the plating tank, the new face actively and uniformly initiates the growth of fresh layers of Ni-P from the bath chemistry. The sputtered seed layer replaces the several steps of zincate activation, presenting to the nickel bath a surface which is identical to the very first layers of Ni-P that result from conventional zincate activation. The layer of sputtered seed Ni-P acts as a sputtered "strike" layer, providing Ni-P available to nucleate new Ni-P layers autocatalytically from the plating bath.

For electroless nickel plating, the pre-treatment of aluminum alloys by double zincate activation is a well-established process.[4,5,6] However, conventional zincate pretreatment can run into problems with composites. By examining individual steps in zincate activation, some new understanding is possible which can extend the uses of electroless nickel. In particular, the present report describes sputtered seed surface activation for plating electroless nickel on materials that do not ordinarily respond to conventional zincate pre-treatment, including silicon carbide and MMC composite alloys of aluminum reinforced with particles of hard compounds such as boron silicide, silicon carbide and aluminum oxide.

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[*] *Corresponding author:*
Dr. Leonard Nanis
LN3 Electrochemical Engineering
2114 Rosswood Drive
San Jose, CA 95124-5427
Phone: (408) 369-1690
E-mail: nanislen@juno.com

Background: MC-21 composite alloys

MC-21 composite material is an MMC alloy with a matrix of type A359 casting alloy (90.4% Al, 9.0% Si, 0.6% Mg) reinforced by micron-sized silicon carbide particles. The SiC content can be readily varied in MC-21 type composites up to 40 vol% SiC.[**]

MC-21 with 30 vol% SiC has useful mechanical properties - a modulus of elasticity twice that of aluminum plus high vibration damping. Applications include body armor and wire-bonding equipment where rapid damping of oscillations minimizes tracking errors. MC-21 is used for heat spreaders in semiconductor packages because of its high thermal conductivity and close match to the coefficient of thermal expansion of silicon. However, early attempts to use MC-21 to replace glass substrates for rigid memory disks stalled because an essential layer of electroless nickel could not be plated without deep pits using conventional zincate activation.

Interest has recently been renewed in MC-21 as a memory disk substrate because its light weight and high stiffness permit desirable rotation speeds upwards of 20,000 RPM, making it a candidate to replace the more fragile glass and distortion-prone type 5586 aluminum alloy substrates.

Background: zincate chemistry

It is generally accepted that the second zincate immersion creates a thin uniform deposit of zinc over the aluminum alloy surface. The thin zinc layer protects against oxidation during transfer of the aluminum to the electroless nickel tank and also serves to nucleate a thin nickel layer by the electrochemical displacement reaction:

$$\mathrm{Zn} + \mathrm{Ni}^{++} \rightarrow \mathrm{Ni} + \mathrm{Zn}^{++} \tag{1}$$

With conventional zincate activation, when the zinc layer on the surface dissolves upon immersion in the Ni-P bath, it is replaced by a nickel layer, according to Equation 1. The nickel layer so generated serves as a catalyst to initiate electroless nickel deposition and layers of Ni-P begin to deposit.

Once initiated, the continuing deposition of Ni-P is considered to be autocatalytic. That is, each deposited layer of Ni-P serves to nucleate the following layer. However, we may ask if the zincate steps are necessary. In other words, can we start Ni-P growth by providing an autocatalytic trigger layer without resorting to conventional wet process zincating to get nickel onto the work surface? The sputter-seeded activation answer is "yes."

As mentioned, one of the advantages of sputter-seeded activation is that it permits Ni-P to be plated on non-metals, such as silicon carbide and glass, as well as on metals which do not respond directly to zincating.

Sputter targets are available in a wide variety of materials to provide binder layers and also seed layers which specifically activate Ni-P deposition.

Sputter-seeded activation has been successfully applied to type 5586 aluminum alloy, widely used for memory disk substrates.[3] The present report describes the use of sputter-seed surface preparation for electroless nickel deposition on less familiar high-strength aluminum based MMC composite alloys.

About the sputter-seed / plate method

Briefly, the sputter-seed / plate method involves only two steps. The first step is vacuum sputtering of a thin seed layer onto a substrate. The second step is conventional electroless nickel plating. Figure 1 shows the main features of the two alternative paths for activation - sputter seed vs. conventional zincate steps. Both paths prepare aluminum 5586 alloy for electroless nickel plating. The sputter-seed method is not limited to aluminum but is applicable, in principle, to any substrate that can be coated by sputtering.

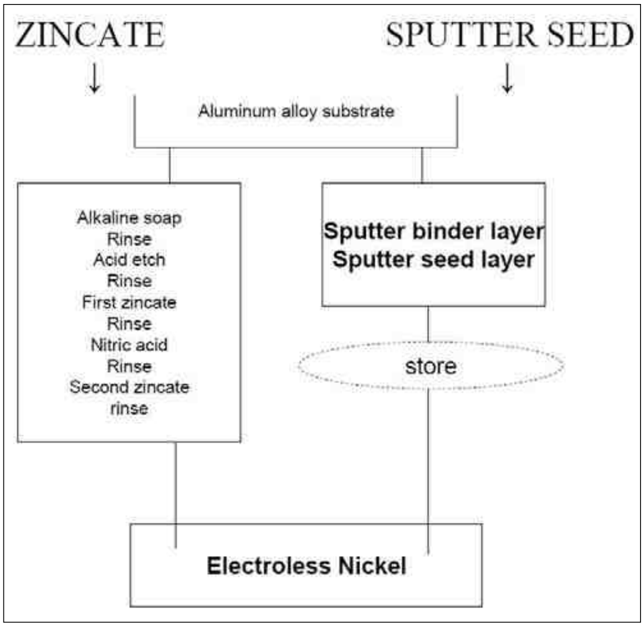

Figure 1—*Comparison of sputter-seed with conventional double zincate activation steps.*

In the sputtering vacuum chamber, an applied voltage ionizes and accelerates gas ions, usually argon, which hit a target material and eject target atoms. The ejected atoms land on a work piece and build up thin films of the target material. The mechanism of sputtering and associated plasma physics is described in the text by Chapman.[7]

To insure adhesion to the substrate, a thin binder layer may first be sputtered onto the substrate and the seed layer then sputtered onto the binder. As shown in Fig. 1, the sputtering of the binder and seed layers takes place sequentially in the same vacuum chamber.

When the thin sputtered seed layer is immersed in the electroless nickel solution, the seed layer instantly nucleates the autocatalytic growth of Ni-P. The growing Ni-P deposit adheres completely to the seed layer. The thickness of binder and seed layers can be as little as 200 to 500 Å, achieved rapidly by sputtering. After the sputter steps, parts removed from the vacuum chamber may be stored in air before plating (Fig. 1), since the seed layer remains active for several days. The convenient extended delay time possible between sputtering and plating is in contrast to the rapid transfer required between final zincate bath and nickel tank, necessary to prevent oxidation of zinc and resulting poor adhesion of Ni-P.

Sputtered binder and seed layers on single-phase alloy

To gain insight into the interaction of the sputter-seed / plate method with composite alloys, it is helpful first to examine the sputtered layers on the nominally single-phase alloy, type 5586 (Al with 4.5% Mg), used primarily as the substrate for rigid thin film memory disks. Transmission electron microscopy (TEM) provides a close look at the sputtered binder and seed layers at high magnification.

Figure 2 shows a view of electroless nickel deposited on 5586 aluminum alloy that had been activated for Ni-P plating by first sputtering a 500-Å chromium binder layer followed by a 500-Å sputtered Ni-P seed layer.

[**] *MC-21 Corp., Carson City, NV; technical reports available at www.mc21inc.com.*

For convenience, thin slices through the layers were rapidly prepared for TEM study by microtoming of the relatively soft alloy. The microtome cut was purposely made at an angle across the layers to differentiate actual structural features from those introduced accidentally by the microtome blade.

In Fig. 2 (650,000×), the sputtered 500-Å-thick layer in contact with the 5586 substrate is identified as chromium by energy dispersive x-ray spectroscopy (EDS) element analysis in the TEM. For clarity, the sputtered layer structure of Fig. 2 is shown schematically in the accompanying Fig. 3. The sputtered chromium binder is a continuous layer composed of individual crystalline grains.

The sputtered seed layer of Ni-P is present above the chromium layer in Fig. 2. The upper surface of the Ni-P sputtered seed layer is at a distance of 500 Å above the top surface of the chromium binder layer. However, there are no features in Fig. 2 to discern a boundary line between the sputtered Ni-P seed layer and subsequently plated electroless nickel. Electroless plated Ni-P merges smoothly with the Ni-P seed layer, indicated schematically by the dotted line in Fig. 3. Seed layer Ni-P and plated Ni-P are both amorphous, as determined by electron diffraction in the TEM. Chromium by itself does not catalyze an adherent layer of Ni-P.

The 5586 alloy substrate sputter seeded with Ni-P shown in Fig. 2 was Ni-P plated to a thickness of 8 μm (315 μ-in.). The as-plated surface of the deposit examined by scanning electron microscopy (SEM-EDS) was smooth, essentially free of nodular growth, reported previously together with details of excellent adhesion.[1,2] A storage period of several days elapsed between seed layer sputtering and Ni-P plating.

It is noted that adhesion of Ni-P plated on aluminum alloy memory disk substrates is customarily tested simply by bending a disk about a mid-line to an angle greater than 90°. No spalling at the bend area signifies good adhesion.

Plating on composite alloys

The particles purposely added to strengthen aluminum-based composites pose a special problem for plating electroless nickel on these alloys. The finely divided particles which contribute strength to the aluminum matrix may be intermetallic compounds or other types of hard compounds such as silicon carbide, boron silicide and aluminum oxide. The hard particles may be chemically inert to conventional wet zincating in the sense that they do not participate in the main reaction of zincating - an electrochemical displacement reaction (Equation 2) where aluminum anodically dissolves as aluminate ion and zinc deposits cathodically:

$$2\ Al + 3\ Zn(OH)_4^{=} \rightarrow 2\ Al\ (OH)_4^{-} + 4\ OH^{-} + 3\ Zn \qquad (2)$$

In MC-21 composite alloys, the matrix alloy (A359) is reinforced by from 10 to 40 vol% of micron-sized particles of silicon carbide. In the conventional zincate pretreatment, SiC particles at the surface are chemically inert, although the matrix does become activated. No growth of Ni-P occurs on the SiC particles since they do not participate directly in the reaction given in Equation 2. Ni-P deposition will start to grow on the activated aluminum alloy matrix but the resulting deposit contains deep pits that originate at non-activated SiC sites.

Ni-P growth may partially fill in the pits by bridging. However, EDS element probing reveals the presence of SiC at the bottom of pits, as in the center of Fig. 5 (to be discussed subsequently).

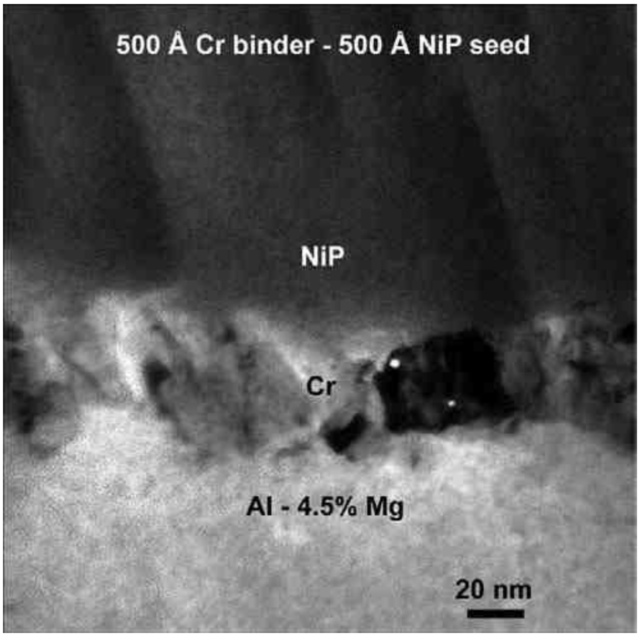

Figure 2—*Sputter-seeded Ni-P on 5586 alloy (TEM).*

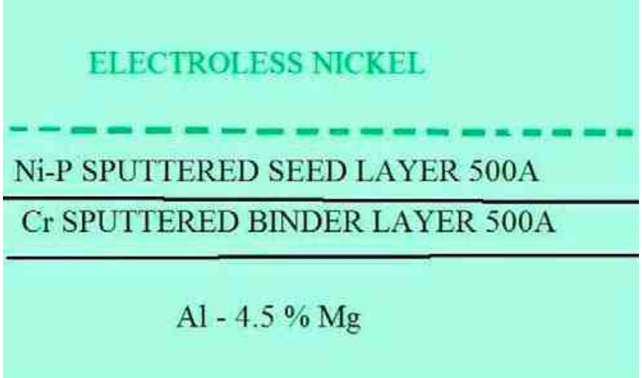

Figure 3—*Schematic arrangement of layers in Fig. 2.*

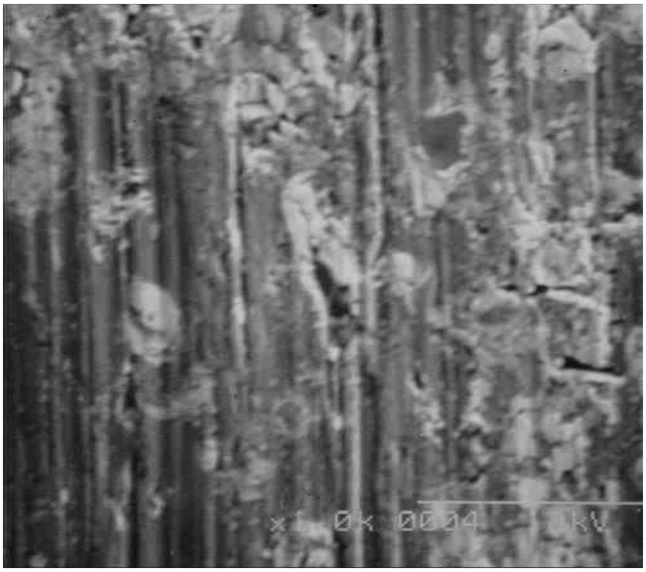

Figure 4—*As-rolled MC-21 sheet (1000×).*

Experimental

The MC-21 composite alloy investigated is A359 aluminum matrix mixed with 30 vol% SiC reinforcing particles.

Two groups of as-rolled MC-21 sheet, 1 mm (0.039 in.) thick, were plated with electroless nickel. One group was activated by a conventional double zincate treatment and the second group was activated by a sputtered seed layer. Each group was Ni-P plated in the same bath.[***] Both zincated and sputter seeded groups were used in the as-rolled condition with no further treatment other than a brief cleaning by wiping with isopropyl alcohol.

Details of steps in the conventional double zincate sequence used with MC-21 are shown below, generally similar to the double zincate sequence used with 5586 alloy, as shown in Fig. 1. Each of the steps listed below is followed by rinsing in deionized water at 25°C.

1. Etch sodium hydroxide, 10 sec.
2. Desmut, 20 sec.
3. Acid, 25 sec.
4. Desmut, 30 sec.
5. First zincate, 30 sec.
6. Acid, 30 sec.
7. Second zincate, 15 sec.

Figure 5—*MC-21, double zincate activation, 1.0-hr Ni-P plate (400×).*

For the sputtered seed group, individual targets positioned in a radio frequency sputtering system[†] provided a first layer of 300 Å of chromium followed by a 300-Å seed layer of nickel-phosphorus alloy. The structure of electroless nickel plated on sputter-seed activated MC-21 was compared with the structure of the control group activated by conventional double zincate treatment sequence, described above.

In addition to the sputter-seeded and conventional zincate groups, a third group of as-rolled MC-21 sheet samples was given a single zincate immersion (only through Step 5), then rinsed in deionized water and dried in clean air with no further treatment. SEM-EDS studies determined the distribution of zinc on MC-21 at the first stage of conventional zincate activation for immersion times of 30 and 180 sec.

Brief experiments were also performed on dense silicon carbide to examine electroless Ni-P plating after sputter-seed activation of SiC sections much larger than the micron-size particles in MC-21. Sputtered and non-sputtered zones on a single flat (4 × 8 × 0.2 cm) section of dense SiC were defined by wrapping part of the area with a removable copper foil mask prior to sputtering. After sputtering (Cr, Ni-P, each 500 Å), the mask was removed before immersing the entire SiC piece in an electroless nickel plating tank for two hours.

Results

Figure 4 (1000×) shows the initial surface of as-rolled sheet MC-21 with 30 vol% SiC particles. The angular particles of SiC (approximately 15 - 20 μm size) are the lighter-appearing phase in the darker shaded aluminum-silicon alloy matrix. A detailed study of the micro-deformation and metallography of MC-21 by Myriounis, *et al.*[8] also shows the presence of silicon and traces of segregated magnesium as magnesium silicide as well as traces of oxygen-containing phases.

In addition to the major phases Al and SiC, Fig. 4 shows parallel marks along the rolling direction. Traces of iron and chromium were detected in the marks, doubtlessly transferred from the alloy steel rolls of the rolling mill. Surface roughening may occur if some of the SiC particles are fractured, detached and re-attached by the roll pressure. The presence of traces of iron and chromium plus silicon, magnesium silicide and traces of oxide phases adds electrochemical complexity in conventional zincate pre-treatment of the surface of as-rolled MC-21.

Figure 5 shows MC-21 after one hour of Ni-P plating following conventional double zincating treatment of as-received, rolled MC-21. The presence of silicon and carbon, detected by EDS probing of the pit near the center of Fig. 5, indicates that a SiC particle at the base of the pit has remained unactivated. As noted, the presence of many such deep pits, visible after polishing, retarded early attempts to introduce MC-21 as a memory disk substrate material.

Sputter-seed activation provides a successful answer to the problem of how to plate defect-free Ni-P on MC-21 for memory disk substrates as well as for other applications. The smooth Ni-P deposit in Fig. 6 was obtained by Ni-P plating catalyzed by sputter-seeded Ni-P and is a distinct improvement over the nodular and pitted deposit shown in Fig. 5, plated after conventional zincate activation. Ni-P plating conditions were identical: one hour at 90°C to obtain a 9.5 μm (375 μ-in.) thick deposit.

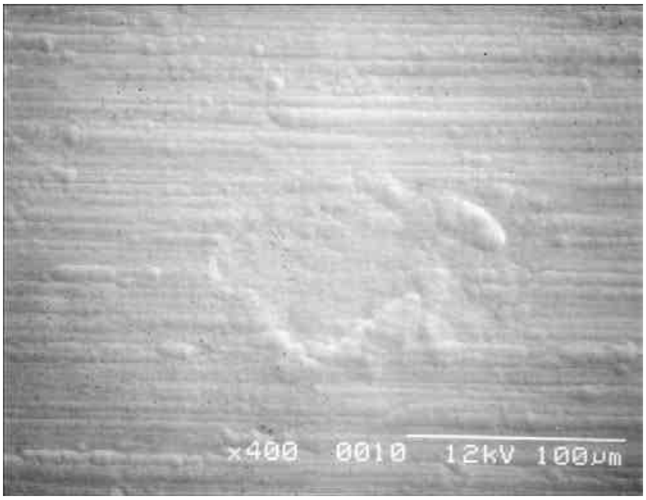

Figure 6—*MC-21, sputter-seed activation, 300 Å Cr, 300 Å Ni-P, 1.0-hr Ni-P plate (400×).*

[***] *OMG Fidelity Hi-Phos, OM Group, Cleveland, OH.*

[†] *SEGI Model FA2-4TR 13.56 megaHertz RF sputtering system, Semiconductor Engineering Group Inc., Milpitas CA.*

The surface in Fig. 6 is essentially free of large nodules and is relatively smooth in comparison to that shown in Fig. 5. As a guess, Ni-P in the middle region of Fig. 5 may be centered on a SiC particle that was sputter-seed activated, permitting deposition.

The linear pattern in Fig. 6 also demonstrates that the Ni-P deposit initiated by the seed layer reproduces the contours of the original surface as it grows. The linear groove pattern in Fig. 6 reproduces traces of the rolling marks shown in Fig. 4, even after a Ni-P thickness of 9.5 μm has been plated. In other words, if you start with a smooth substrate, sputter-seeded electroless nickel plates to a smooth finish, since there is no etching and roughening of the original surface as produced with conventional alkaline zincate activation.

The greatly reduced amount of nodular Ni-P growth possible with sputter-seeded activation simplifies polishing, if required after plating, as for the critically smooth finish on memory disks substrates. Less polishing means cost savings because of higher throughput, fewer polishing steps as well as reduced amounts of polishing materials, process water and waste disposal.

The changes of the initial as-rolled surface during conventional zincate activation steps are of interest, particularly the behavior of the silicon carbide reinforcing particles. Surface appearance was examined for single zincate immersion, Steps 1 to 5, listed previously. As shown in Fig. 7, the single zincate produces significant changes compared with the as-rolled MC-21 surface (Fig. 4).

In general, 30 sec of zincate immersion etches and further roughens the as-rolled surface. In Fig. 7, the A359 aluminum matrix has the appearance of a mesh network, suggesting chemical attack during the single zincate treatment. The A359 matrix receives a coating of zinc even as it becomes textured in the mesh network pattern. While it is not certain exactly which of the five steps produces the greatest change, it most likely occurs in the highly alkaline zincate solution.

From EDS elemental analysis, the prominent angular particles in the central region of Fig. 7 are silicon carbide, whereas zinc and silicon are in the smaller, slightly rounded, light colored particles. The key result from the single zincate study is the complete absence of zinc on SiC particles in the MC-21 microstructure.

As shown in Fig. 7, SiC particles do not participate in activation during 30 sec of first zincate immersion, which helps to explain the source of deep pits in Ni-P plated on MC-21 activated by conventional double zincate treatment. It is unlikely that an acid dip and second zincate immersion will improve plated Ni-P coverage. Studies of structural changes made by the individual immersion steps of zincate activation remain as an area for future investigation.

Silicon carbide particles (dark areas in Fig. 8, 1500×) still remain free of zinc after an increase in first zincate immersion time to 180 sec. Additional deposition of zinc occurs with apparent smoothing of the deposit on the etched A359 matrix aluminum. A high concentration of magnesium and silicon (presumably Mg_2Si) was detected at the center of the cluster of zinc-rich small rounded particles near the lower right corner of the large SiC grain (upper center) in Fig. 8. As mentioned, Myriounis, *et al*.[8] identified the presence of Mg_2Si as a minor phase in a metallographic study of the structure of polished hot-rolled MC-21 with 31 vol% SiC.

The zinc coating on the matrix increases in thickness after 180 sec of first zincate immersion, as indicated qualitatively by an increased ratio of zinc to aluminum EDS intensity peaks. Zinc added during 180 sec (Fig. 8) appears to fill in the mesh structure obtained at 30 sec (Fig. 7). As noted, for both immersion times, particles of SiC stand out prominently, without zinc on their surfaces.

We may consider the absence of zinc on SiC particles as a reasonable cause for the pits present in plated Ni-P on MC-21 activated by conventional zincating. Although only the surfaces resulting from first zincate immersion up to 180 sec were examined, it may be expected that longer first zincate immersion plus a nitric acid dip and brief second zincate immersion would not add zinc to the SiC particles. As noted, visualization of the effect of individual immersion steps remains as an area for future investigation.

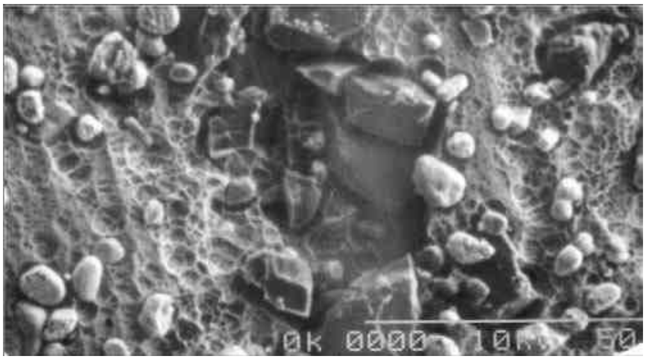

Figure 7—*MC-21, 30 sec first zincate (1000×).*

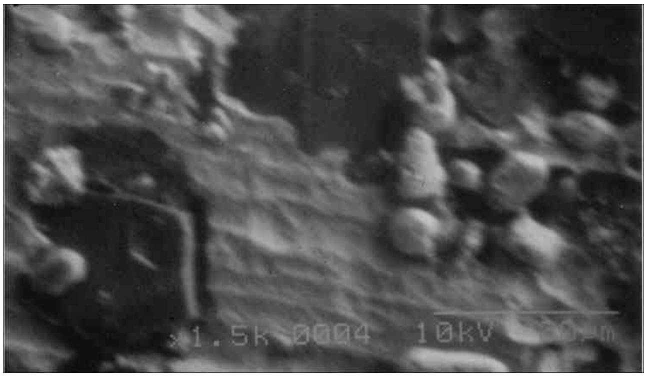

Figure 8—*MC-21, 180 sec first zincate (1500×).*

Brief experiments performed with dense pure SiC sheet sputtered with chromium binder and Ni-P seed layers (except for a masked zone) yielded a completely adherent deposit of Ni-P on the sputter-seed activated zone. Plating initiated on the sputter-seeded zone, whereas the adjacent zone that had been masked remained unplated. There was a sharp dividing line between plated and unplated zones, with no lateral transfer of Ni-P growth.

From this brief auxiliary study and the evidence from SEM examination of zincated and also plated MC-21 (Fig. 5), it may be concluded that SiC does not catalytically nucleate Ni-P deposition, either by itself or when in close proximity to regions undergoing Ni-P deposition.

Discussion and conclusions

The evidence from this study points to silicon carbide particles as the site of deep pits in electroless nickel, activated by conventional zincate preparation of MC-21 composite alloy. It is also clear that sputter-seeded activation can compensate for the complexity associated with wet chemical treatment of multi-phase MMC alloys.

With some development effort, perhaps the chemistry of zincate activation can be modified to accommodate MC-21 (and similar MMC alloys) so as to avoid pits. Additional studies to visualize the MC-21 surface and to pinpoint the effects of all phases present as it progresses through the individual steps of zincate pre-treatment may be helpful in adjusting the chemistry. However, sputter seeded activation presents a clear advantage by covering all phases in the MC-21 surface, *i.e*., the Al-Si alloy matrix, SiC particles, minor phases Si and Mg_2Si, as well as trace oxide phases and trace impurities of iron and chromium transferred by rolling.

The experience with Ni-P plating on purposely added SiC particles may also be related to the behavior of intermetallic particles normally present in aluminum alloys, especially if they do not participate in the zincate activation. Much metallurgical effort has been devoted to reducing the size of intermetallic particles in aluminum alloys in order to reduce the number of defects in Ni-P plated on memory disk substrates. The sputter-seed method is an alternative approach to minimizing the effect of intermetallic and other types of inclusions in other types of alloys. Rather than seeking to modify the size and distribution of particles, sputter-seed activation accepts the particles as they are and adds a thin layer to cover over all separate phases of the alloy. Also covered are elements and compounds inadvertently transferred to the work surface by mechanical means, such as rolling and polishing.

Sputtering (PVD) bypasses conventional zincate activation by depositing the seed layer and binder layer as thin films. The outer surface of the seed layer serves essentially as a pre-existing layer of Ni-P, ready to nucleate succeeding layers when immersed in the plating tank. Basically, the seed layer is a sputtered “strike,” ready to jump-start autocatalytic Ni-P deposition.

For some applications, sputter-seeded activation can play a significant role, especially if the material to be Ni-P plated does not respond to conventional zincating. In the case of glass or ceramic materials, sputter-seeded activation can replace activation by palladium - tin immersion methods.

Workpieces that are nearly planar (*e.g*., memory disk substrates, mirrors, cooling surfaces) fit readily in several commercially available sputtering systems. Systems with increased size vacuum chambers are also available.

Selective plating by Ni-P can be obtained by masking the part with metal foil patterns or by photolithographic techniques before sputtering so that only the area designated for plating receives a sputtered seed layer.

From a practical cost benefit viewpoint, the cost of a sputter system has to be weighed against the cost of a multi-tank plating line. Likewise, the cost of consumable targets has to be compared with the cost of various chemicals in the zincate line, their storage, clean water requirements for the makeup of solutions, analysis and control of baths, rinse water and waste disposal. Tank and chemical costs for electroless nickel are basically the same for each method of activation although longer life may be expected for a dedicated electroless nickel solution that receives only sputter-seeded parts, since there is no accumulation of zinc. In the cost trade-off between sputter-seed and conventional zincate, throughput of each method must also be considered.

The resulting smooth surface of the Ni-P deposit can be a distinct advantage, particularly if post-plate polishing can be minimized. In principle, a deposit of Ni-P plated on an initially smooth surface should be just as smooth. As mentioned, the sputter-seed method is not limited to aluminum alloys. Metals or compounds which do not participate chemically in conventional zincate reactions can be sputter-seed activated for Ni-P plating. Sputter-seeding has also been used successfully to plate adherent Ni-P on glass, nylon, pure silicon carbide shapes and on composite alloys of aluminum strengthened by boron silicide nanoparticles. As MMC composite alloys find new applications that also require a coating of electroless nickel, sputter-seed is ready.

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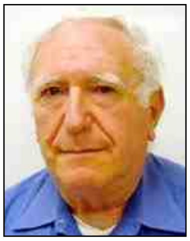
About the author



Dr. Leonard Nanis is founder and President of his own consulting firm, LN3 Electrochemical Engineering. He is also Consulting Professor in the Department of Materials Science and Engineering at Stanford University. Dr. Nanis holds B.S. and M.S. degrees in physical metallurgy from MIT, where he worked with Prof. Carl Wagner. He received his Doctorate of Engineering Science in extractive metallurgy at Columbia University with Prof. H.H. Kellogg. Post-doctoral work followed at the University of Pennsylvania where he served as Assistant Director of the Electrochemistry Group headed by Prof. John O'M. Bockris.

Dr. Nanis was Professor of Chemical Engineering at the University of Pennsylvania where he taught courses in heat and mass transfer, fluid mechanics, applied mathematics and electrochemical engineering and directed research in battery technology, molten salt chemistry, electrode kinetics, corrosion, hydrogen embrittlement and current distribution. After several years at Penn, Dr. Nanis went on to direct research on advanced batteries, fuel cells and low-cost extractive metallurgy of silicon for solar cells at SRI International, Menlo Park CA.

Dr. Nanis was Chairman of the Industrial Electrolytics Division of the Electrochemical Society from 1980 to 1982. He received the ASTM Templin Award in 1970 and, in 1998, an award from Hyomen Gijutsu (J. Surf. Fin. Soc. Japan).