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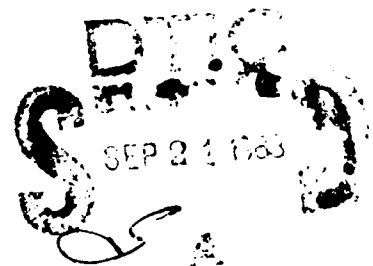# DEPARTMENT OF MATERIALS AND METALLURGICAL ENGINEERING

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PULSED ELECTRODEPOSITION OF AMORPHOUS AND COMPOSITE ALLOYS

FINAL REPORT

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)
Novel uses of pulsed current in the electrodeposition of alloys were investigated. The study resulted in the demonstration of the feasibility of depositing alternate layers of the same phase or different phases of controlled chemical compositions and thicknesses. The alternate-layer structure resulted in better strength properties in some cases. An orders-of-magnitude increase in the ductility in some layered alloys was discovered. Pulse plating also reduced uncontrolled compositional variations which cause the banded structure.

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8

Table of Content

List of Illustrations and Tables

INTRODUCTION

The objective of this investigation was to study the methods by which pulse plating could improve the properties of electro-deposits by altering the chemical-composition vs thickness profile. This use of pulse plating differs considerably from the way it is presently applied which is primarily to enable plating at high current densities without the usual accompanying detrimental structures. The alterations of the composition-thickness profile which were investigated were both the enhancement of composition variations and their elimination.

The enhancement of chemical-composition variations with thickness involved the deposition of composition-modulated structures. These structures consisted of alternate layers of various predetermined thicknesses of the same thermodynamic phase but of different chemical composition i.e., a structure analogous to that produced after spinodal decomposition. The investigation of the enhancement of the composition variation also included the deposition of alternate layers of two different phases i.e., a structure analogous to lamellar-eutectic composites. A new phenomenon was discovered that deposits consisting of alternate layers of the same phase and essentially the same chemical composition but having different grain sizes produced by different pulses, exhibited unusually high ductilities.

The elimination or reduction of variations in the chemical composition with deposit thickness was also accomplished. Some alloys when plated under conventional conditions, exhibit the so-called banded structure which results from uncontrolled compositional variations with thickness. Pulse plating results in a more uniform composition in these alloys. The alloy systems which were used in the investigations of the enhancement and the elimination of the compositional variations were Ni-Mo, Cu-Zn and Ni-P.

STUDIES OF PULSE-PLATED Ni-Mo ALLOYS

Electrodeposition of Ni-Mo alloys is of scientific interest because molybdenum by itself cannot be plated from aqueous solutions. The alloys also possess good corrosion-resistance properties as well as high strengths. In this study the alloys were investigated in connection with the enhancement of composition-thickness variations, i.e., the deposition of alternate layers of two different molybdenum contents. The compositions of the several plating solutions which were used in the investigations are listed in Table 1.

Deposits from the baths listed in Table 1 all exhibit very high internal stresses when plated with plain direct current. Therefore the investigation had to consist initially of finding ways to reduce the stresses. It was further necessary that the remaining internal stress be as uniform throughout the thickness as possible in order to obtain deposits of adequate thickness which would not disintegrate when their substrates were dissolved. The instantaneous internal stresses were determined during the deposi' using the microbalance device described by Feigenbaum and Weil.[1] Mo alloys were deposited on electropolished copper-sheet substrates in Bath A at 60°C at

current densities of 2.5, 5, 10 and 20 mA/cm^2. The stresses did not vary significantly with thickness during the first 1 µm. After this thickness, the stresses decreased slowly. The stresses in the first 1 µm are plotted vs current density in Fig. 1. It is seen that the stresses in deposits plated with plain dc, i.e., no pulse are quite high and increase significantly with increasing current density. The application of a pulse with an on time equivalent to 20 mC(millicoulombs)/cm^2 (i.e., current density x time) and a 10 sec. off time resulted in a considerable stress reduction especially of the highest stresses. The pulse was selected because an earlier study[2] of stress indicated that the coalescence of crystallites is a primary cause of stress. The crystallite size of the Ni-Mo deposits was determined by transmission electron microscopy to range from 5-25 nm in the plane parallel to the surface. Choi and Weil[3] found that nickel crystallites have essentially the same dimensions perpendicular to the surface as parallel. Passing 20 mC/cm^2 results in the deposition of approximately one layer of crystallites. Souzis and Weil[4] found that stress relaxation took place in a few seconds after the current is turned off. Thus the stresses should relax during the 10 sec. off time. The results shown in Figure 1 appear to corroborate that a considerable fraction of the total internal stress is due to crystallite coalescence. This fraction apparently was reduced by allowing the coalescence stresses to relax during the off time before a new layer of crystallites was laid down. However it is also apparent from Figure 1 that there still remains a considerable stress which was not due to crystallite coalescence.

Hydrogen is believed to be another cause of internal stress in Ni-Mo deposits. Hydrogen which is codeposited on the surface can diffuse into the deposit resulting in a shrinkage of the surface layer and a tensile stress. Using a pulse with an on time equivalent to 2 mC/cm^2 results in the deposition of approximately one monolayer of atoms. The hydrogen adsorbed on the surface could desorb during the off time and not diffuse inward. Analysis for hydrogen of the plain dc and the pulse-plated sample with the greatest stress, i.e., the one plated at 20 mA/cm^2 by SIMS showed a reduction from 350 counts to 200 counts. So far it has not been possible to obtain standards to translate these numbers into percentages. As is apparent from Figure 1, the higher frequency pulse resulted in a further stress reduction and more importantly, the stress no longer varies significantly with current density.

The molybdenum contents of deposits plated with the 2mC/cm^2 on and 1 sec. off pulse varied from about 5% by weight when the current density was 2.5 mA/cm^2 to about 10% when it was 25 mA/cm^2. Deposits with higher molybdenum contents were too brittle for mechanical testing. Originally the molybdenum contents were reported to be higher because the standards for the flourescent X-ray analysis were not correctly chemically analyzed.

The mechanical properties of Ni-Mo deposits plated at 2.5 mA/cm^2 and at 25 mA/cm^2 with the 2mC/cm^2 on and 1 sec. off pulse and also of composition-modulated structure were determined. Three types of tensile tests were conducted. Bulge tests of 5 µm thick samples and

uniaxial tensile tests in an Instron machine of 10 μm thick samples were conducted. The samples were deposited at 2.5 mA/cm^2 and at 25 mA/cm^2. In addition there were composition-modulated deposits tested consisting of alternate layers. A 0.8 μm thick layer was deposited at 2.5 mA/cm^2 and a 0.2 μm thick layer deposited at 25 mA/cm^2. The three different kinds of samples were tested in the as-plated condition and after annealing at 300°C. The reason for the 300°C anneal will be discussed later. From the results listed in Table 2 it is seen that the fracture strengths of the modulated samples are higher than those of the ones plated at only one current density. The fracture strengths obtained by bulge testing and in the Instron machine agree well. The lower elongations obtained by bulge testing are attributable to the state of biaxial stress which prevents necking. Samples 1 μm thick were also tested in which the layers were one-tenth the thickness, i.e., those plated at 2.5 mA/cm^2 were 0.08 μm thick and those plated at 25 mA/cm^2 were 0.02 μm thick. These samples were tested in a mini-tensile machine described in a recent paper.[5] The modulated deposits annealed at 300°C were considerably stronger as seen in Table 2. Their higher fracture strength is probably due to the smaller spacing of the alternate layers. The higher strength of the samples tested in the mini-tensile machine as compared to those tested in the Instron or by bulging is attributable to several factors, namely the smaller specimen dimensions[5], the fact that the specimens were not cut to size and better gripping and aligment. It is apparent from Table 2 that the modulated structures do not show improved ductility. However, some composition-modulated deposits exhibited very high ductilities with elongations exceeding 100%. This phenomenon will be discussed in connection with the Cu-Zn alloys.

The recrystallization kinetics of the Ni-Mo alloys were investigated in order to find the proper recovery temperature for stress relieval. This work was carried out by the apprentice high school student during the Summer 1982 under the US Army Research Office sponsored program administered by the Academy of Applied Science. The proper recovery temperature was found to be 300°C. Samples plated at 2.5 mA/cm^2 were annealed in vacuum at several temperatures after dissolving their copper substrates. They were then thinned for transmission-electron-microscopic examination. The percent of the area which had recrystallized was determined on photographs using a planimeter. From a graph of percent recrystallized volume assuming equiaxed grains vs reciprocal absolute temperature, activation energies of 20.2 and 28.2 kJ/mole were determined for the sample plated at 2.5 and 25 mA/cm^2, respectively.

From Dr. Ahmed of Watervliet Arsenal it was learned that amorphous Ni-Mo deposit can be obtained from Bath B in Table 1. Experiments leading to the possible deposition of alternate layers of amorphous and crystalline layers were started. Samples were plated at 2.5, 25 and 100 mA/cm^2 at 25°C and 60°C. They were chemically analyzed and using transmission electron microscopy it was determined whether they were amorphous. Samples plated at the three current densities with plain dc at 25°C had Mo contents of 40, 30 and 16%, respectively. All were amorphous. The deposits were so brittle that they disintegrated into small pieces, which

were fortunately still big enough for transmission-electron-microscopic examination, when their substrates were dissolved. Pulse plating had relatively little effect on the chemical composition, the amorphousness or the brittleness. The same experiments were carried out with modifications of Bath B, called Baths C and D in Table 1. The conditions for depositing alternate layers of amorphous and crystalline Ni-Mo alloys have not yet been determined. It is expected that work towards this goal will be continued.

STUDIES OF PULSE-PLATED Ni-P ALLOYS

It has been known for a relatively long time that metal glasses can be produced by electrodeposition or autocatalytic chemical reduction (often called electroless plating). Materials so produced have the advantage over those made by rapid cooling from the melt in that they can be produced at ambient temperature and do not have the shape or thickness limitations. However, the mechanical properties especially the ductility and corrosion resistance of the plated metal glasses are inferior to those of the rapidly cooled ones. The plated metal glasses when their cross sections are examined metallographically exhibit the so-called banded structure. There are alternate layers with thicknesses in the order of microns, which have been found to be an etching effect due to composition variations. Figure 2 shows the structure seen by scanning electron microscopy with the accompanying energy-dispersive-X-ray-analysis profile of the phosphorus content of a plated Ni-P alloys. There were some indications that the banded structure was at least partially responsible for the inferior properties of the plated metal glasses.

Studies were conducted to try to eliminate by pulse plating the compositional variations with thickness responsible for the banded structure. Using a plating solution composed of 30 g/ℓ NiSO$_4$ x 6 H$_2$0, 10 g/ℓ sodium hypophosphite and 10 g/ℓ sodium acetate, experiments were conducted at various current densities, pulse frequencies and duty cycles (the ratio of the on time to the on plus off times) and pH values. It was found that the band spacing increased and the difference in the phosphorus content between the darker and lighter bands decreased with pulse plating. Also the overall phosphorus content increased with pulse plating. Although the internal stresses were not very high, pulse plating resulted in a reduction. The optimum conditions for elimination of the compositional variations were 100 mA/cm^2 with a 1 msec on and 1 msec off time. Some samples plated with this pulse still showed faint bands which could be brought out only by special etching techniques. The energy-dispersive-X-ray profile however showed no phosphorus variations greater than background noise. To date, the virtual elimination of the compositional variations has not led to a significant improvement of the mechanical properties.

It is now believed that the inferior properties of the plated glasses are mostly due to hydrogen embrittlement. Samples plated with plain d.c and pulse plated under the optimum condition previously described were submitted to Prof. Lanford of the State University of New York at Albany for the determination of hydrogen content vs thickness profiles. The first attempts were unsuccessful because of

apparently large amounts of hydrogen present near or on the surface. Another set of samples which was recently sent will be held at very low temperatures. The results of these tests are not yet available. It is apparent that more work is needed to assess the application of pulse plating to the metal glasses.

STUDIES OF PULSE PLATED Cu-Zn ALLOYS

In order to demonstrate the feasibility of electrodepositing structures analogous to lamellar-eutectic composites, i.e., consisting of alternate layers of two different phases, Cu-Zn alloys were chosen. The main reason for this selection was that the intermetallic phase, β brass has a relatively simple crystallographic structure, namely body-centered cubic in the disordered state and a low atom ratio, one copper to one zinc atom. Intermetallic phases with higher ratios, e.g., the phase Ni_4Mo require considerable surface diffusion of atoms to form in the correct stochiometry, which is apparently not possible during electrodeposition. When such a phase cannot crystallize, alloys of its composition deposit in the amorphous state.

Experiments to determine the conditions for depositing the terminal solid solution, α brass and the intermetallic β phase were conducted in plating solutions of several compositions. The bath which gave the best results contained 32 g/ℓ CuCN, 55 g/ℓ ZnCN, 95 g/ℓ NaCN, 20 g/ℓ Na_2CO_3 and 20 g/ℓ NH_4OH. The pH was adjusted to 10.2 by additions of $NaHCO_3$. The bath temperature was 36°C. The composition of the deposits with various pulses as a function of current density were determined for example, it was found that β phase of 52% Cu can be deposited at 7 mA/cm^2 with 1 msec on and 1 msec off pulse and at 100 mA/cm^2 with 1 msec on and 5 msec off. Plating at 3 mA/cm^2 with 1 msec on and 1 msec off or at 100 mA/cm^2 with 1 msec on and 1 msec off yields α solid solutions of 65% Cu. These four conditions were selected for further investigation primarily because only 2 different pulses namely 1 msec on, 1 msec off and 1 msec on, 5 msec off were involved. In this way, the programming was simplified.

Studies to determine the minimum thicknesses of the two phases which could be deposited were conducted. Layers of different thicknesses of one phase sandwiched between layers of the other phase were plated and examined by transmission electron diffraction to determine if the two crystal structures were present. Layers of either phase as thin as 50 nm were identified without difficulty. The orientation relationships between the phases were also determined from the electron diffraction pattern for thin layers and for thick ones by X-ray diffraction. Attempts to determine the composition variation at the phase boundaries by sputtering off layers and analyzing them by Auger electron spectroscopy were unsuccessful probably because of the high vapor pressure of zinc.

Deposits consisting or alternate layers of α and β phases of various thickness were produced. Figure 3 shows the cross section of a deposit consisting of alternate layers of α and β brass plated at the lower current densities of 3 and 7 mA/cm^2, respectively and at 100 mA/cu^2 with the two pulses. Figure 3 shows that all these structures can be plated in the same bath. The layers pictured in Figure 3 were plated thick so that the composition of each could

be determined by energy-dispersive-X-ray analysis. The α brass was found to be 71.0 atomic % Cu and the β was 51.4 atomic % in the layers plated at the lower current density. In the layers plated at 100 mA/cm^2 the α phase contained 64.0 atomic % Cu and the β had 51.8 atomic %.

The mechanical properties of 10 μm thick deposits of α and β which were deposited separately and having alternate layers of the two phases were determined by bulge testing. The results are shown in Table 3. It is seen that in general the two-phase deposits have poorer fracture strengths than the single-phase ones and about the same ductilities. It is probable that another alloy system having an intermetallic phase of much higher strength than β brass would show an improvement in properties for deposits having structures analogous to lamellar-eutectic composites. It may be recalled that the Cu-Zn system was chosen primarily to show the feasibility of depositing alternate layers of two phases which was accomplished.

The phenomenon of high ductilities previously found in some Ni-Mo alloys was observed also in deposits consisting of alternate layers of the same phase, the β phase deposited at 7 mA/cu^2 with the 1 msec on and 1 msec off pulse and at 100 mA/cu^2 with the 1 msec on and 5 msec off pulse. To date the phenomenum has not been observed in deposits consisting of alternate layers of the α phase deposited at the two current densities respectively or alternate layers of both phases. Results of bulge tests of 10 μm thick deposits of several different layer thicknesses are shown in Table 3. The deposit consisting of 0.5 μm and 0.25 μm thick layers plated at 7 and 100 mA/cu^2, respectively shows the best ductility. It is probable that this alloy would show ever high ductility in a uni-axial tensile test. Tests of samples of other thickness combinations are planned.

An initial investigation to elucidate the phenomenon was con-ducted. Transmission-electron-microscopic examination of a sample thinned by electropolishing the 10 μm thick deposit plated at 100 mA/cu^2 showed a relatively large grain size of the order of 200-500 nm in diameter. The deposit plated at 7 mA/cu^2 when examined in the same way had a much smaller crystallite size of the order of 50 nm and showed a very strong texture. The deposit con-sisting of alternate layers which has the high ductility shows crystallites with sizes again of the order of 50 nm. The selected-area-electron diffraction pattern of these crystallites was essentially that of a single crystal with a <111> direction per-pendicular to the surface. As the samples for transmission-electron-microscopic (TEM) examination were thinned by electropolishing, it is not known which layer was being observed. Even by examining several samples, which was done, one is not certain that both layers would be examined as one may be electropolished away preferentially. Attempts to produce samples for TEM examinations of foils which are thin perpendicular to the surface have not yet been successful. Scanning-electron-microscopic examination of the cross section show a uniform structure. Apparently the high-ductility phenomenon is associated with a combination of a very small crystallite size and a high degree of texture. There are reports in the literature[6] of superplastic behavior at room temperature in alloys with very small grains in which the texture was produced by plastic deformation.

It appears that investigators in the Soviet Union are also interested in the electrodeposition of layers by pulse plating as indicated in a recent paper[7] entitled "Current Yield and Mechanical Properties Deposited in Pulsed Conditions". The closing sentence of the English translation is "Moreover, promise may be shown by layerwise deposition at different frequencies, as this should yield cathodic deposits with different physical properties in the layers".

PRINCIPAL ACCOMPLISHMENTS

1. The feasibility of depositing alternate layers of the same phase of different chemical composition by the use of pulse plating was demonstrated. The compositions and layer thicknesses could be controlled over a range of predetermined conditions. After stress-relief annealing, the fracture strength of layered deposits was greater than that of the non-layered types.

2. The feasibility of electrodepositing alternate layers of two different phases by the use of pulse plating was demonstrated. In the alloy system selected for this demonstration, namely Cu-Zn, the two-phase composite deposits did not possess better mechanical properties than single-phase ones.

3. Deposits consisting of certain thicknesses of alternate layers of the same phase electroplated with different pulses were discovered to have orders-of-magnitude increases in ductility.

4. The feasibility of using pulse plating to obtain a more uniform chemical composition-vs-thickness profile in alloys exhibiting the so-called banded structure, which is caused by a non-uniform composition, was demonstrated.

5. A reduction in internal stresses was attained by appropriate pulses. Information about the causes of these stresses was obtained from the pulse characteristics.

Clearly, further investigations of the demostrated novel uses of pulsed electrodeposition are indicated. Especially for Accomplishment No. 3, an understanding of the involved mechanisms is needed to determine its applicability to alloys other than those studied and to optimize the ductility of electrodeposits. The proposal, which has resulted in continued support by the Army Research Office, which is gratefully acknowledged, indicated the direction of the further studies.

A statement of invention has been submitted to the Patent Committee of Stevens Institute as a first step in obtaining a patent dealing with the layered deposits.

PUBLICATIONS AND AWARDED DEGREES

Two papers were presented at the Fall 1981 meeting of The Electrochemical Society. The extended abstracts were published in the proceedings of this meeting. A paper was also presented

at the Winter 1982 meeting of the Metallurgical Society of AIME. Several papers for publication are in the process of preparation. As soon as some loose ends are tied down, Mr. C. C. Nee expects to present this work as his doctoral thesis to the faculty of Stevens Institute of Technology.

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Table 1

Compositions of Ni-Mo Plating Solutions

	Bath A	Bath B	Bath C	Bath D
$NiSO_4 \times 6\ H_2O$	84 g/ℓ	48 g/ℓ	96 g/ℓ	96 g/ℓ
$Na_2MoO_4 \times 2\ H_2O$	7-9 g/ℓ	48 g/ℓ	48 g/ℓ	48 g/ℓ
Sodium Citrate	72 g/ℓ	105 g/ℓ	105 g/ℓ	105 g/ℓ
$NaHCO_3$	—	—	—	—
pH	10.5	10.2		10.5

Table 2

Mechanical Propeties of Ni-Mo Alloys

	Samples Plated at 2.5 mA/cm^2		Samples Plated at 25 mA/cm^2		Composition-Modulated Samples	
	σ_f*	e_f**	σ_f*	e_f**	σ_f*	e_f**
Bulge Test						
As Plated	38	0.5	24	0.4	41	0.8
300°C anneal	60	0.8	76	0.5	86	1.6
Instron Test						
300° Anneal	51	5.6	71	6.4	88	1.0
Mini-Tensile Test						
As Plated	92	1.0	230	2.7	130	1.7
300°C anneal	130	1.4	190	2.5	340	5.0

* Fracture Strength in Kpsi
** Fracture Strain in percent

Table 3

Mechanical Properties of Cu-Zn Alloys

Single Phase Alloys

Plating Conditions				True Fracture	True Fracture
Current Density (mA/cm^2)	On Time (msec)	Off Time (msec)	Phase	Strength (kpsi*)	Strain (%)
3	1	1	α	26	5
7	1	1	β	70	4
100	1	1	α	65	2
100	1	5	β	65	4

Alternate Layers of Two Phases

Layer Thicknesses (μm)		True Fracture	True Fracture
α**	β***	Strength (kpsi)	Strain (%)
1	1	28	4
0.5	0.25	23	5
0.25	0.12	22	8
0.1	0.05	25	3

Layers of Beta

Layer Thicknesses (μm) Deposited at		True Fracture	True Fracture
7 mA/cm^2	100 mA/cm^2	Strength (kspi)	Strain (%)
0.25	0.12	53	11
0.5	0.12	65	15
0.5	0.25	77	27

  * 1 kpsi equals approximately 7 megapascals
 ** deposited at 3 mA/cm^2
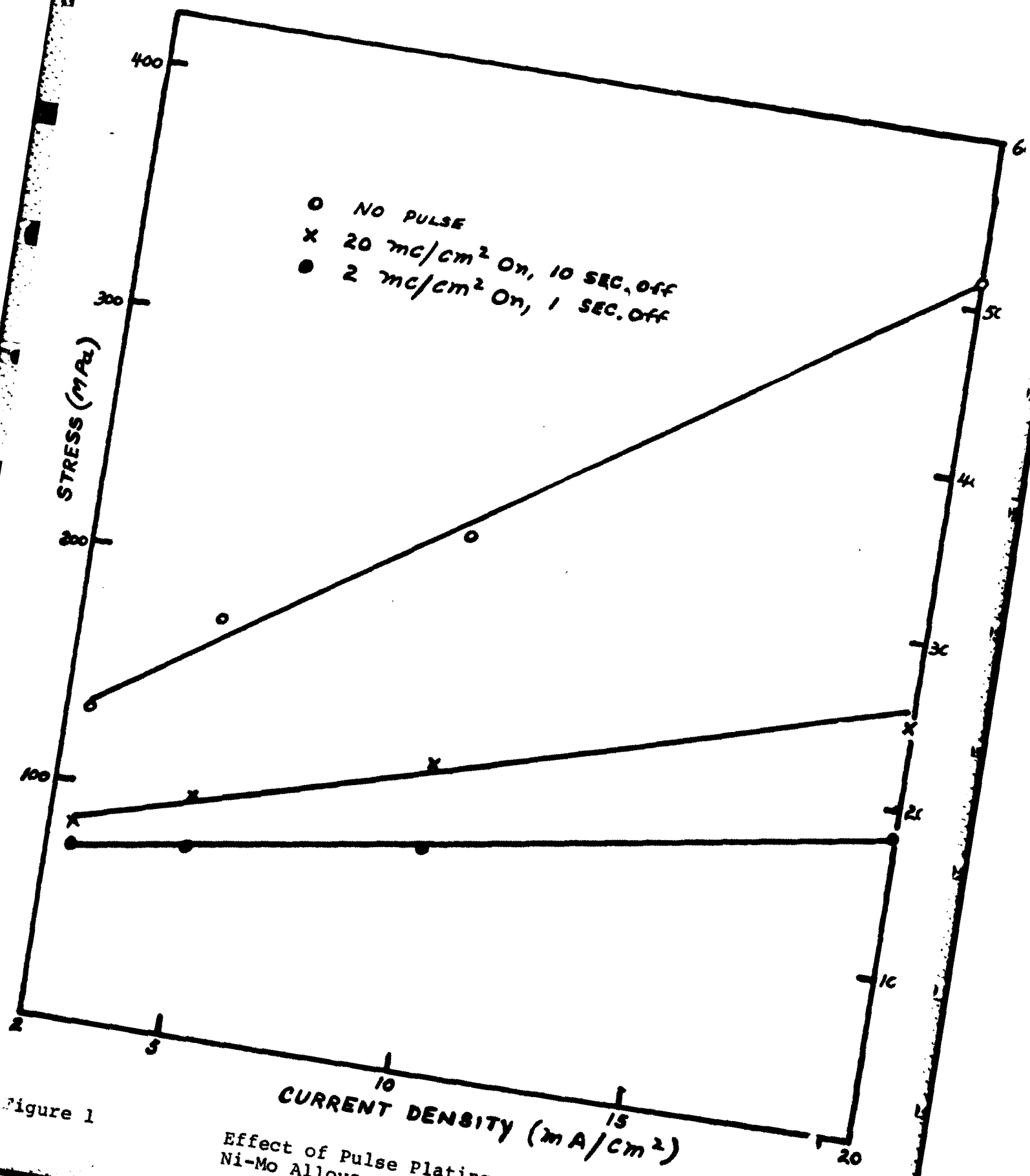*** deposited at 7 mA/cm^2

Figure 1

Effect of Pulse Plating on the Internal Stresses of Ni-Mo Alloys.
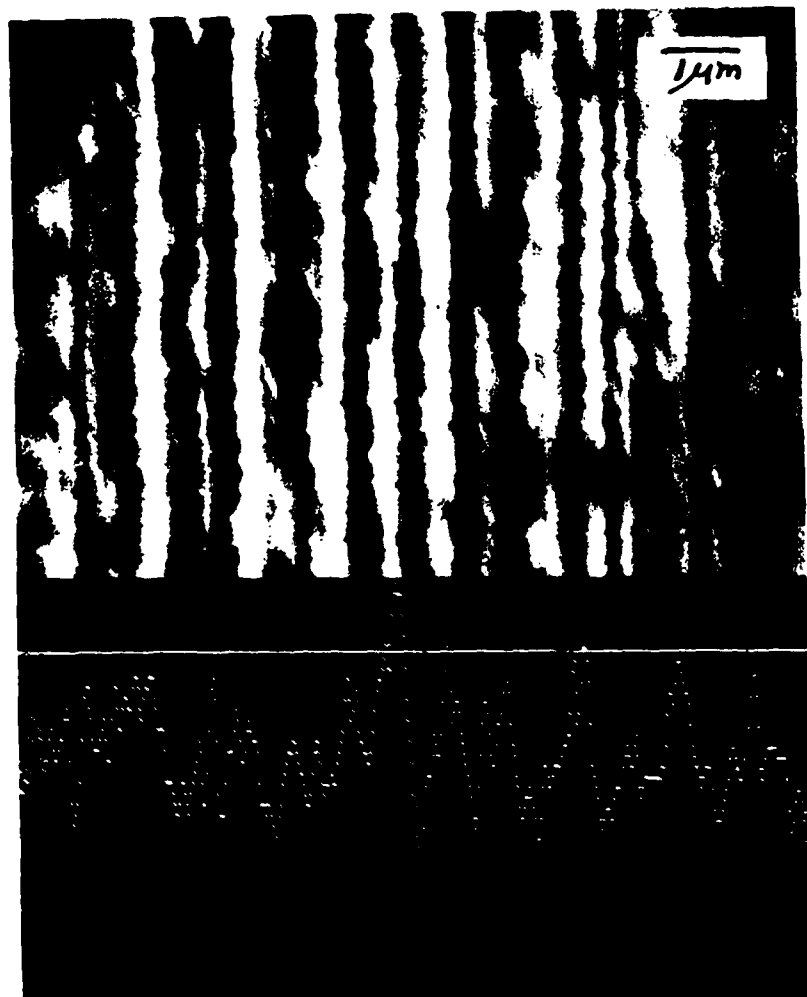
Figure 2   Scanning Electron Micrograph
of Banded Structure of Ni-P Alloy and
Corresponding Phosphorus Content as
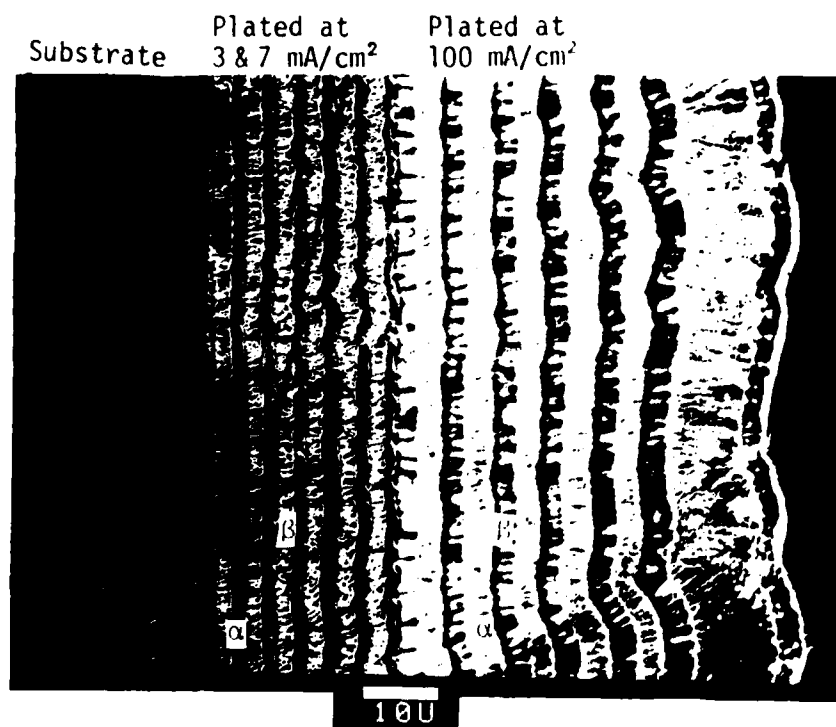Determined by Energy-Dispersive-X-Ray
Analysis.

Figure 3. Scanning Electron Micrograph of Cross
Section of Cu-Zn Alloy Showing α and β Layers. First
Set Pulse Plated at Low Current Density of 3 mA/cm²
and 7 mA/cm² Respectively. Second Set Pulse Plated
at 100 mA/cm².

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