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Techniques for Quantitatively Measuring Adhesion of Electrodeposits

(To be presented at *SAMPE "Technology in Transition" Symposium* to be held in San Diego in April 1975)

MASTER

J. W. Dini, H. R. Johnson

Sandia Laboratories

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TECHNIQUES FOR QUANTITATIVELY MEASURING ADHESION OF ELECTRODEPOSITS

J. W. Dini
H. R. Johnson
Metallurgy and Electroplating Division 8312
Sandia Laboratories, Livermore

ABSTRACT

Five techniques for quantitatively testing the adhesion of electrodeposits are reviewed: ring shear, conical-head tensile, modified Ollard, I-beam, and flyer plate. The ring shear and conical-head tensile tests are self-explanatory by definition alone. Modified Ollard and I-beam are tensile-type tests and flyer plate tests are shock wave tests used for evaluating parts under dynamic loading conditions. All have been shown to be quite effective in providing consistently reproducible data. Details are presented for each test including shape and size of specimens, preparation of specimens, and manner in which the test is performed. Cost comparisons and facility requirements are also defined, and data for various substrate-electrodeposit combinations are included.

ACKNOWLEDGMENTS

Thousands of plated parts have been tested by using the techniques described in this report. To do this required considerable help from the staff at SLL, including plating, machining, testing, and analytical support. We thank all who have been actively engaged at some time or other in the work, particularly J. R. Helms, plating; W. Young, machining; J. Jost, cost estimating and machining; A. Clark, S. Grisby, W. B. Vandermolen, J. Totten, R. Jacobson, testing; and T. L. Bryant, metallography. We would also like to acknowledge Peter Dean for his assistance in the preparation of this report.

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TECHNIQUES FOR QUANTITATIVELY MEASURING ADHESION OF ELECTRODEPOSITS

Introduction

The importance of adhesion* between electrodeposited metals and their respective substrates has increased with the trend toward more engineering applications of plated coatings. In many cases, the qualitative adhesion tests previously used, such as tape, bending, and heating are no longer adequate and must be replaced with tests that produce quantitative data. In view of this need, a number of comprehensive reviews(1-3) have been made, and the Electrochemical Society sponsored a symposium in October 1974 on the subject.

Sandia Laboratories, Livermore presently uses five techniques to quantitatively assess the adhesion of coatings: ring shear, conical-head tensile, I-beam, modified Ollard, and flyer plate shock tests. The conical head, I-beam, and modified Ollard apply a tensile force to the coating-substrate composite, the ring shear applies a shear stress, and the flyer plate test provides a dynamic test of the coating-substrate composite. The ring shear tests have been used more often than the rest of the tests primarily because of ease of obtaining specimens for testing and lower cost of sample fabrication. The I-beam tests, a recent innovation, offer promise for use as an inexpensive screening-type test and will undoubtedly find increased usage. The conical-head tensile tests fill a distinct need wherein tensile properties of the plated system are needed. For situations wherein coated parts will be exposed to dynamic loading conditions, the flyer plate tests are ideal for evaluation purposes.

In order that these five Sandia quantitative test methods can be compared with methods previously described,(1-3) details are presented in

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*Adhesion refers to the bond (chemical and/or mechanical) between two adjacent metals and is measured by the force required to effect their complete separation.

this paper for each test including shape and size of specimens, preparation of specimens, and manner in which the test is performed. Cost comparison and facility requirements are also defined, and data for various substrate-electrodeposit combinations included.

Ring Shear Tests

Zmihorski[4] originally used a ring shear test to measure adhesion between electrodeposited chromium and steel. A cylindrical rod was coated with separate rings of chromium of predetermined widths and from 0.203 to 0.610 mm (8 to 24 mils) thick. This rod was then forced through a hardened steel die that had a hole larger in diameter than the rod but smaller than that of the rod plus coating. The coating was detached and the adhesion strength R was determined from the formula

$$R = \frac{p}{\pi ds} = \mathrm{MN/m^2} \text{ or psi}$$

where p = load to cause failure

d = diameter of cylindrical rod

s = width of chromium deposit

This test was elaborated upon by Mockus,[5] who experimented with many different substrates and platings. Recently, Dini and Johnson[6, 7] published detailed data on the use of this test.

Test Procedure

Most of the work done at Sandia Laboratories has been with rods 12.7 mm (1/2 inch) in diameter and 305 to 381 mm (12 to 15 inches) long. Rods of this diameter are used because they are quite often an "off-the-shelf" item for many materials, thus eliminating the need for any machining prior to plating. Typically, five 25.4 mm (one-inch) segments are stopped off with platers' tape or surgical tubing (see Figure 1) so that five separate specimens can be machined from the rod, thereby offering the possibility of obtaining more than one piece of data for each test condition. The rod is coated with a deposit about 1.52 mm (60 mils) thick, and then a ring 1.59 mm (1/16 inch) thick is machined axially in each plated segment (Figure 2). In cases where precious metal platings such as gold or silver are being evaluated, only enough metal (normally 0.076 to 0.102 mm (3 to 4 mils)) is plated to assure a diameter greater than that of the die's opening. The rest of the

Figure 1. Stainless Steel Rod Before and After Plating

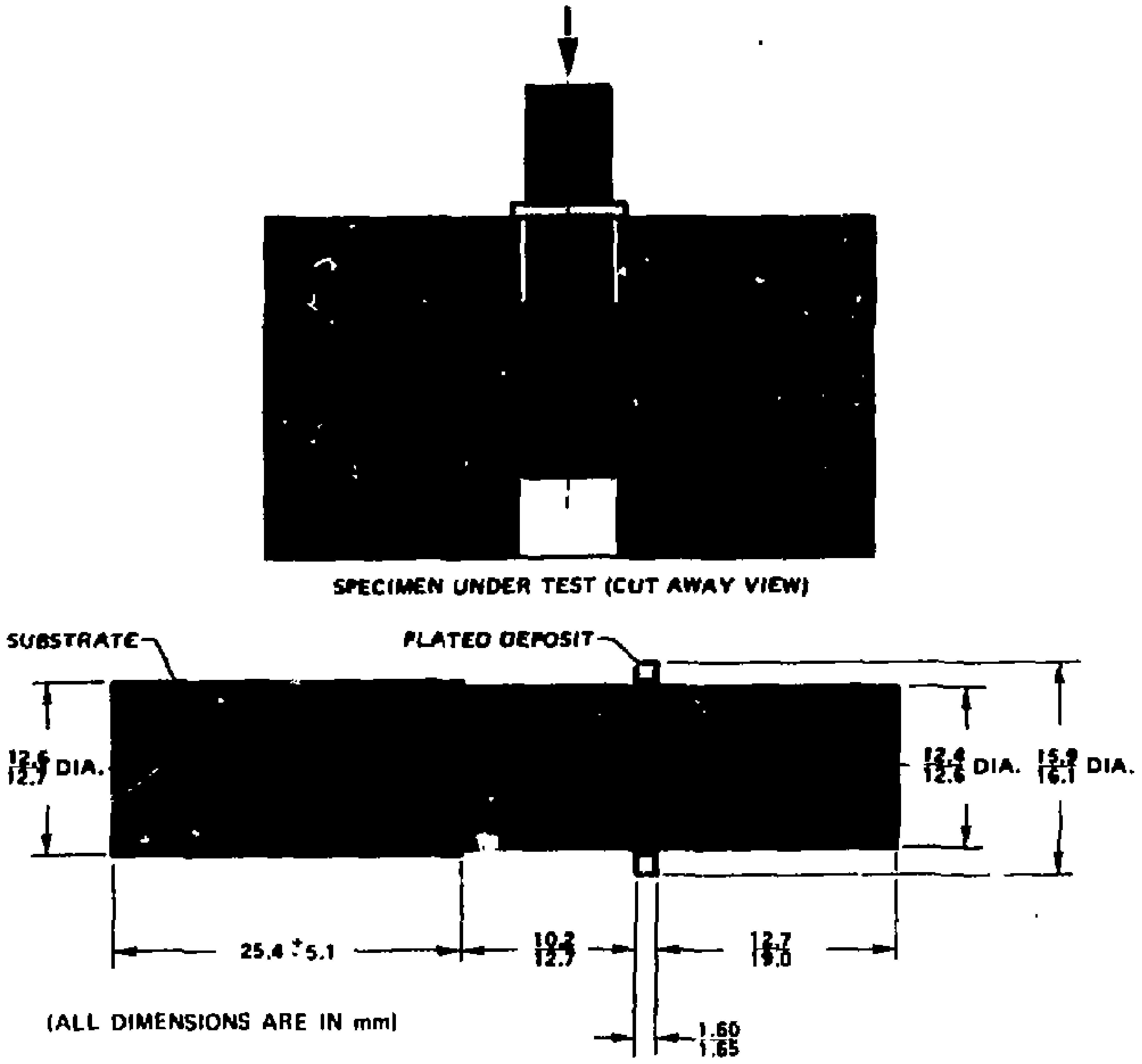


Figure 2. Ring Shear Test Specimen

ring is then plated with either copper or nickel up to 1.52 mm (60 mils) thick. This procedure not only reduces expense, but in the case of gold also shortens plating time since copper or nickel can be plated at much faster rates than gold.

A machined sample and the die used for testing are illustrated in Figure 3. Best results are obtained when the die diameter is 0.051 to 0.102 mm (2 to 4 mils) larger than the rod diameter. The die was made of 4340 steel, heat treated to a hardness of Rockwell C58. This material or another high-strength steel should be used to avoid any danger of distorting the die surface, particularly near the shearing edge.

If 12.7 mm (1/2 inch) diameter rods or material from which to machine them are not available, smaller diameter rod and dies can be used. At Sandia, rods as small as 5.35 mm (1/4 inch) in diameter have been used.

A laboratory hand-operated press can be used for testing specimens; however, best results are obtained with a Universal Testing Machine (Instron). The Instron allows for exact determination of failure, whereas the accuracy of the hand press suffers due to both the deposit yielding during loading and the imprecision of the gages. After testing, the failures can be located visually or with metallographic techniques. When adhesion is poor, the deposit separates from the substrate at the interface of the two; when adhesion is good, separation occurs within either the deposit or substrate (see Figure 3).

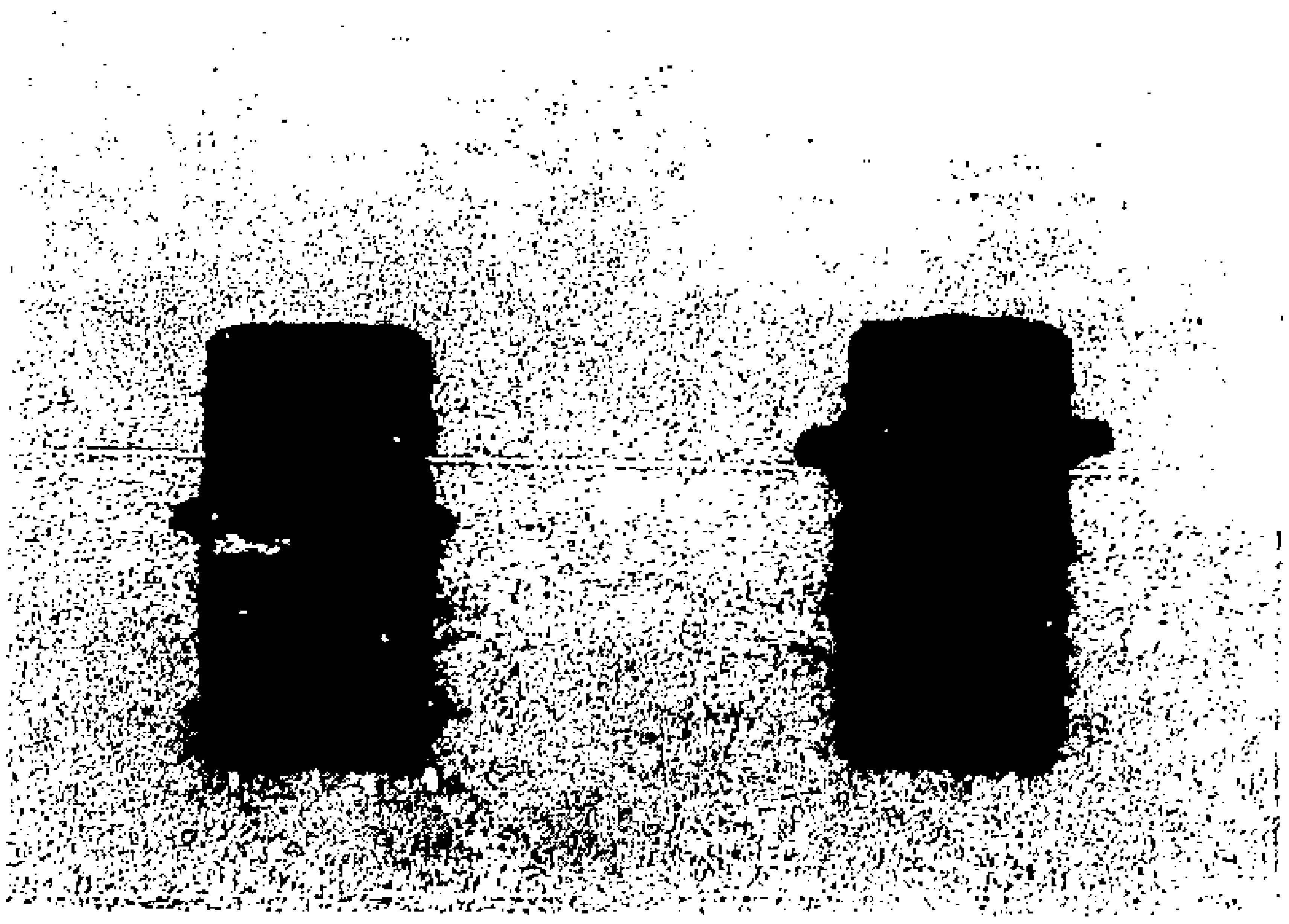


Figure 3. Ring Shear Test Specimens
Left--good adhesion (failure occurred within plated deposit)
Right--poor adhesion (failure occurred at substrate/deposit interface)

Typical Applications

References 6 and 7 contain numerous examples of ring shear test results. For illustrative purposes, two tests are discussed here: the influence of current density during Wood's striking[8] on the subsequent adhesion between nickel and AM363 stainless steel, and the influence of temperature on the bond strength between plated nickel and uranium.

Table I shows the influence of the current density during Wood's nickel striking. Poor adhesion was obtained at 162 A/m^2 (15 A/ft^2) or less in the Wood's strike; strength levels were about 55 MN/m^2 (8000 psi) and less, and failure occurred at the nickel-stainless steel interface. Quite satisfactory adhesion (approximately 324 MN/m^2 (47,000 psi)) was obtained at Wood's current densities of 270 and 540 A/m^2 (25 and 50 A/ft^2); however, even better adhesion was evident at 1080 A/m^2 (100 A/ft^2) and higher. Shear strength at these levels was about 483 MN/m^2 (70,000 psi).

Table II shows the effect of heating on the bond strength of nickel electroplated uranium. The average bond strength of unheated nickel-plated uranium as measured by the ring shear test was 254 MN/m^2 (36,800 psi). Samples heated at temperatures up to 316C for one hour before testing at room temperature showed no reduction in bond strength. At 371C, a gradual reduction was noted such that samples heated at 482C failed at 175 MN/m^2 (25,400 psi); however, even this failure level represented a respectable bond strength. Figure 4 is a cross section typical of the samples heated at 427C for one hour before testing. A number of locations at the interface where failure occurred during testing show evidence of tearing in the nickel deposit, while the remainder of the failure occurred at the nickel-uranium interface. The spaces (black areas) between the nickel and uranium occurred because of the difficulty in repositioning the test ring on the substrate from which it was sheared. These spaces are not voids which were in the original sample or present after heating.

Figure 4. Cross Section of Shear Test Sample Heated at 427°C for One Hour Before Testing (Original magnification 75X)

TABLE I

ADHESION OF NICKEL PLATED ON AM363 STAINLESS STEEL AFTER NICKEL STRIKING(A)

Current Density in Wood's Nickel Strike		Ring Shear Strength(B)		Location of Failure
A/m^2	A/ft^2	Mn/m^2	psi	
54	5	48	6,900	Nickel-stainless steel interface
162	15	54	7,800	Nickel-stainless steel interface
270	25	318	46,100	Greater than 80% within nickel deposit
540	50	337	48,900	Greater than 80% within nickel deposit
1280	100	488	70,700	Within nickel deposit
1555	144	457	66,300	Within nickel deposit

(A) The cleaning/plating cycle consisted of anodic treatment at 324 A/m^2 (30 A/ft^2) for two minutes on Oakite 90 solution, rinsing, immersion in 18% (by weight) HCl for two minutes, rinsing, Wood's nickel striking (240 g/l nickel chloride $6H_2O$, 120 ml/l HCl) for two minutes, rinsing, and plating in nickel sulfamate solution at 270 A/m^2 (25 A/ft^2).

(B) Each reported value is the average of five tests.

TABLE II

EFFECT OF HEAT TREATING FOR ONE HOUR AT VARIOUS TEMPERATURES ON THE BOND STRENGTH OF NICKEL-PLATED URANIUM(A)

Heat Treat Temperature(B) (°C)	Ring Shear Strength(C) (MN/m^2)	(psi)
None	254	36,800
38	257	37,200
93	247	35,800
149	237	34,400
204	261	37,800
260	304	44,100
316	279	40,000
371	234	33,900
427	215	31,200
482	175	25,400

(A) All samples were etched in a solution containing 512 g/l $NiCl_2$, $6H_2O$, and 340 ml/l HNO_3 prior to plating with thick nickel in sulfamate solution.

(B) Heating was done in air.

(C) Average of three samples were all tested at room temperature.

Conical-Head Tensile Tests

With this test, developed by Moeller and Schuler,[9] the electrodeposit, the substrate, and the bond between the two are tested in a tensile fashion, the bond being normal to the loading direction. The specimen design and a pictorial sketch of a part under test are shown in Figure 5. This test which uses standard tensile testing methods is the best technique for quantitative testing of plated bond in a truly tensile manner. For this reason it is probably a better measure of bond strength than any of the other static tests described in this report. As an example, substrates that are heavily etched before plating typically exhibit high ring shear bond strengths when subjected to ring shear tests. However, conical-head tensile tests with the same type of substrate quickly reveal whether the bond is metallurgical in nature or simply mechanical.

Typical Applications

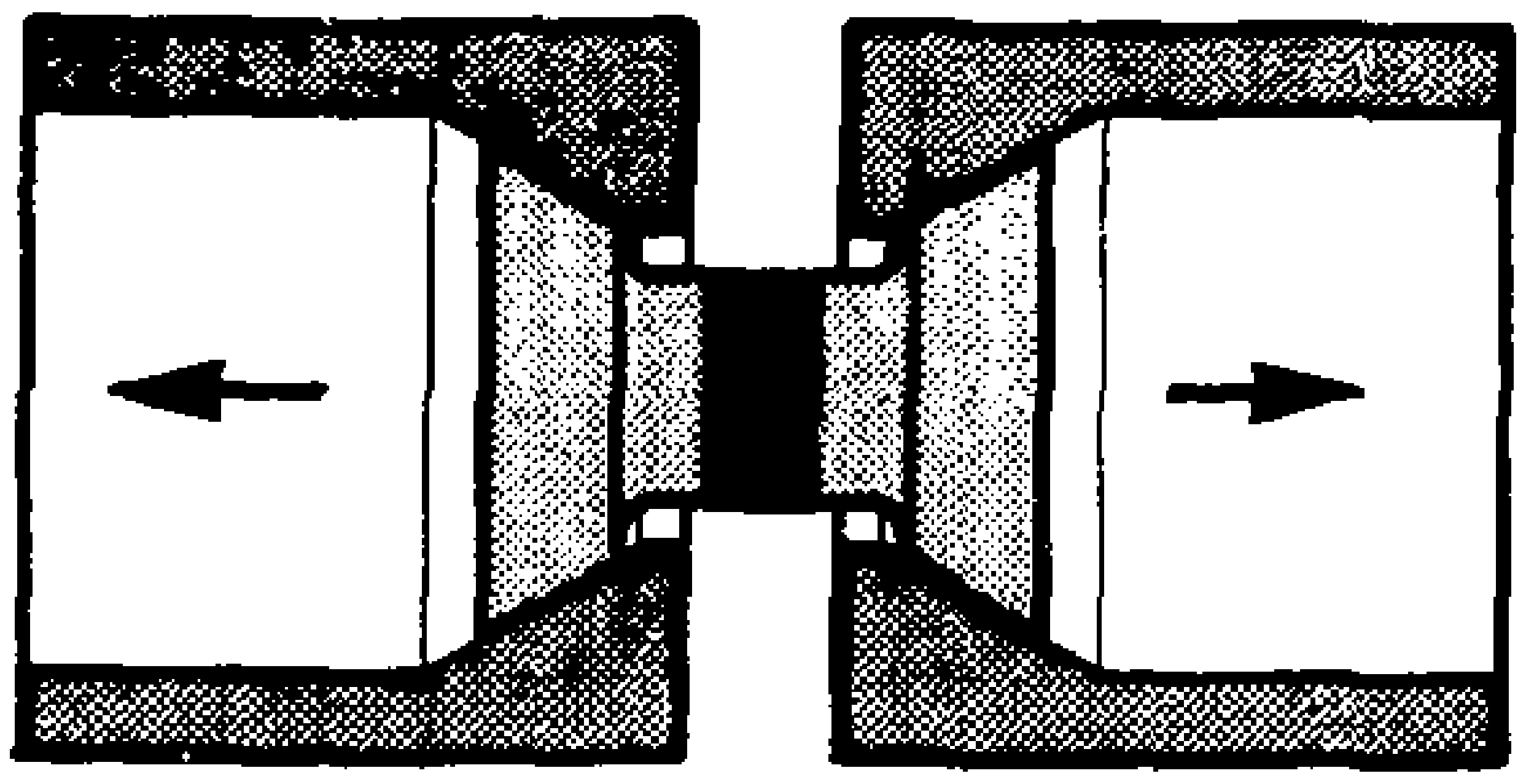
The conical-head test can be used in many applications. For example:

- Tensile and reduction in area data can be obtained for electroformed specimens in the transverse direction. The property data obtained with other test methods is usually associated with a loading direction parallel to the plating substrate rather than perpendicular.

- The influence of temperature upon the properties of solid electroforms or the bond between deposits and substrates can be evaluated. Moeller and Schuler[9] clearly showed the value of conical-head tensile testing when checking bond strength and properties of copper and nickel deposits over the temperature range of -200 to 1832C.

To illustrate that the conical-head tests do subject the electrodeposit bond to a true tensile force, adhesion results are presented in Tables III and IV and Figures 6 and 7 for electroplated beryllium and aluminum. As shown in Figure 6, conical-head tests of nickel-plated ingot-grade beryllium produced a failure in the beryllium. Similar tests of copper-plated P-1 grade beryllium, which is considerably stronger than ingot-grade beryllium, caused failure in the copper-plated section of the test specimen (see Table III).

Data for 2024 and 7075 Al showed that in all cases failure occurred in the aluminum. Figure 7 is a cross section showing the appearance of a 7075 Al sample after testing. The failure in the Al is clearly evident; no damage is seen in the nickel plating or at the interface between the plating and the aluminum. With the 7075, some reduction in conical-head tensile strength was noted for samples heated at 316 C (600°F) for two hours. This was a loss in strength of the aluminum itself; not a reduction in the strength of the bond between the aluminum and the plating.



SPECIMEN UNDER TEST (CUT AWAY VIEW)

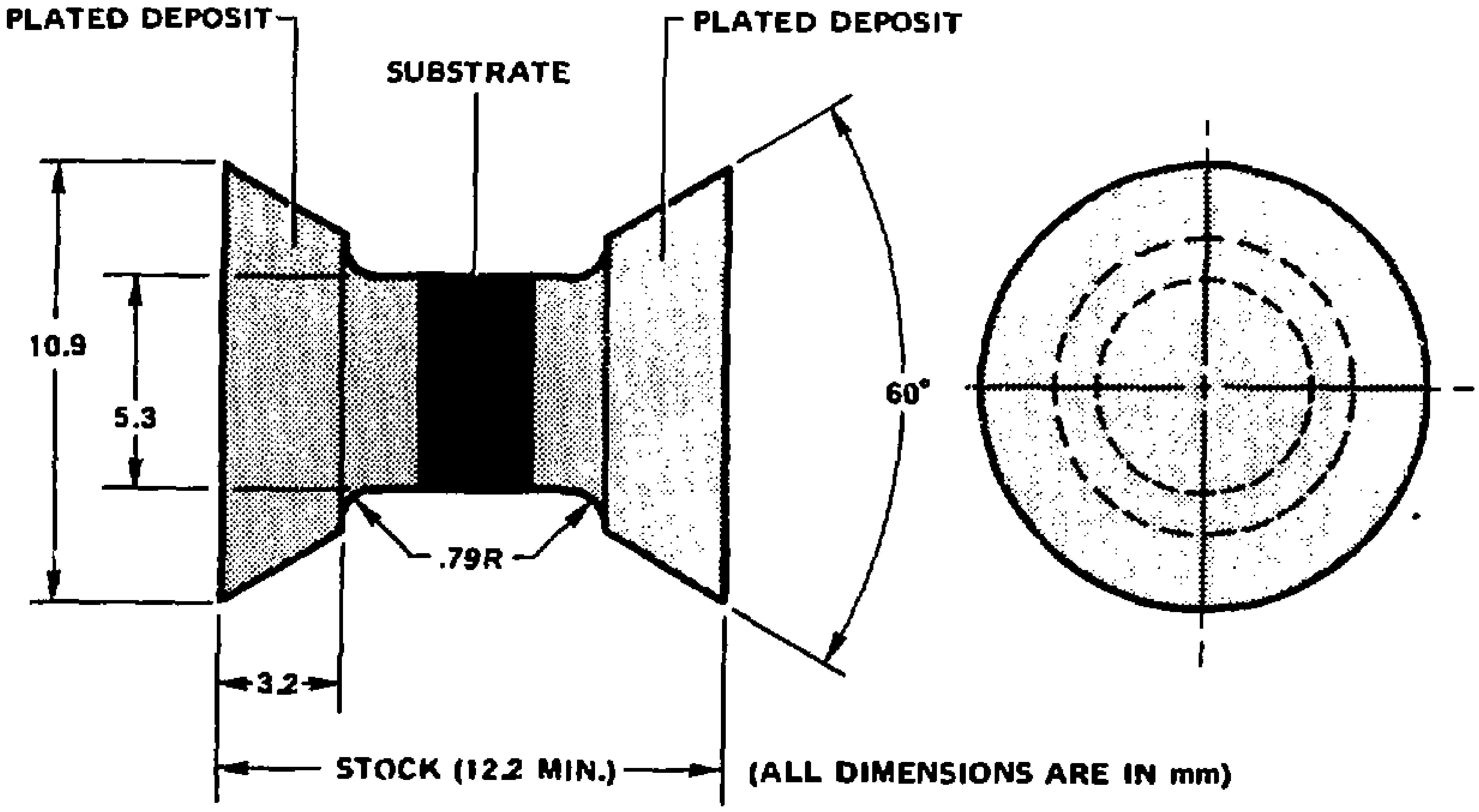


Figure 5. Conical-Head Tensile Specimen (from Reference 9)
(All dimensions are in mm)

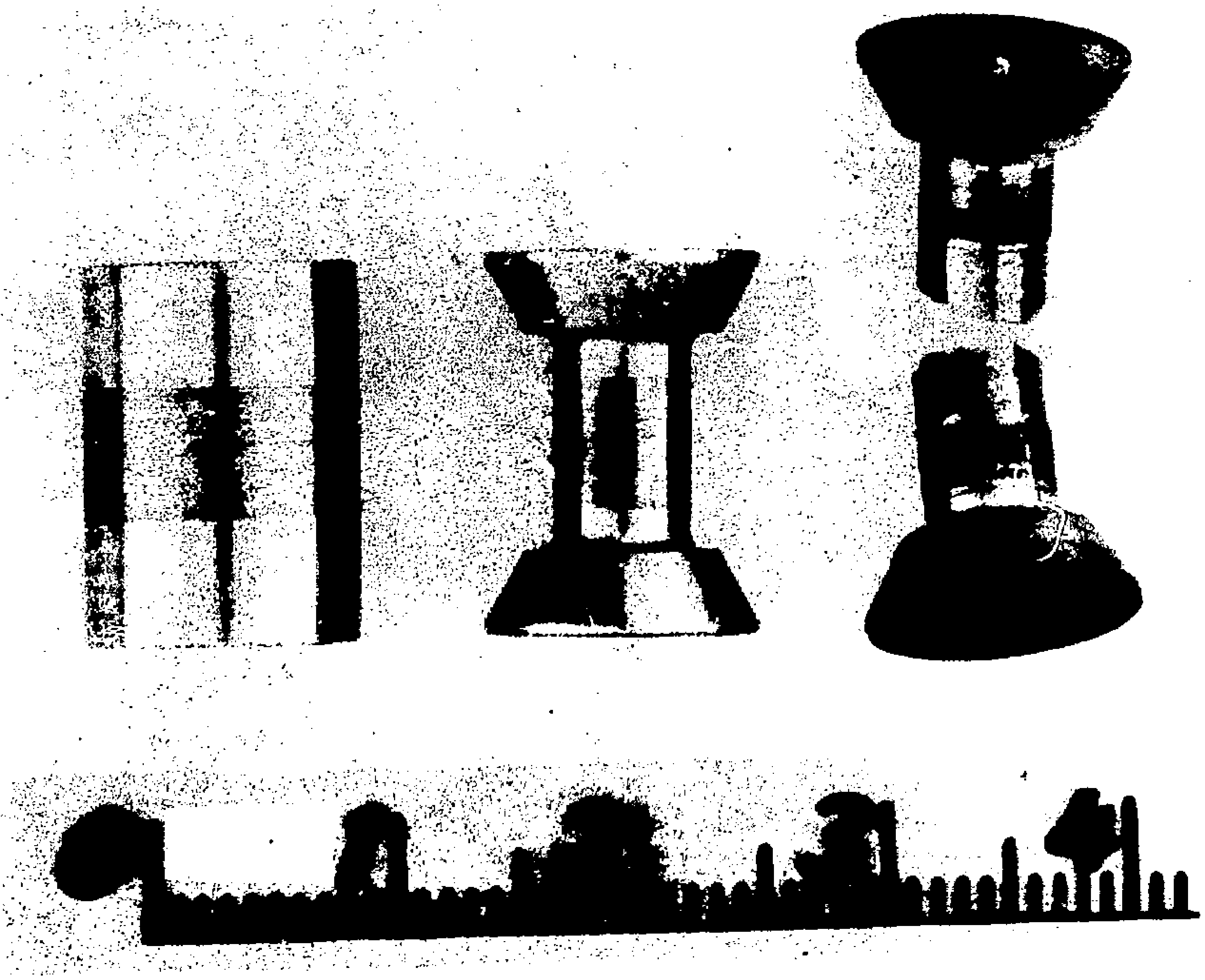


Figure 6. Conical-Head Tensile Specimen of Nickel Plated Beryllium Before and After Testing

Figure 7. Cross Section of Nickel Plated 7075 Aluminum Conical-Head Tensile Specimen After Testing (Magnified 6X)

TABLE III

CONICAL-HEAD TENSILE DATA FOR ELECTROPLATED BERYLLIUM(A)

Beryllium(B)	Type of Plating	Tensile Strength (MN/m^2)	(psi)	Location of Failure
Ingot	Nickel	172	24,900	Be
P-1	Copper	321	46,500	Cu
(After Heating at 316 C (600 F) for Four Hours)(C)				
Ingot	Nickel	168	24,400	Be

(A) The beryllium was prepared for plating by the following process: clean, acid etch, zincate, copper strike, and then final plating per above. For more detailed information, see Reference 10.

(B) Approximately 0.2 mm (8 mils) of Be was removed by chemical milling before tensile testing.

(C) Testing was done at room temperature.

TABLE IV

CONICAL-HEAD TENSILE DATA FOR NICKEL-PLATED ALUMINUM(A)

Aluminum	Treatment(B)	Tensile Strength (MN/m²)	(psi)	Location of Failure
2024-T4	As-deposited	445	64,500	Al
2024-T4	Heated at 149C for 75 mins	428	62,100	Al
7075-T6	As-deposited	572	83,000	Al
7075-T6	Heated at 149C for 1 hr	572	82,900	Al
7075-T6	Heated at 316C for 2 hrs	416	60,300	Al

(A) The aluminum was prepared for plating by the following process: clean, nitric pickle, zincate, nitric pickle, zincate, copper strike, and then final plating per above.

(B) All testing was done at room temperature.

Modified Ollard Test

The Ollard[11] test measures the adhesion of a plated coating by pulling the coating directly from its substrate in a tensile fashion. Because the original technique was only applicable to 25.4 mm (one-inch) diameter rods, Knapp[12] modified it for sheet materials. With either the rod or sheet material, the tensile load is supplied by a tensile testing machine, and the adhesion is calculated from the known failure load and the area of the bond. Information on standardization of the test, such as effect of die clearance, thickness of deposit required, and effect of annular test section height, is given in Reference 12. To reduce sample preparation time, Rothschild[13] utilized a high speed silver plating solution and specially designed drills instead of machining on a lathe.

The slightly modified version of Knapp's technique shown in Figures 8 and 9 is used at Sandia. The difference between this technique and that of Knapp's is that only one side of the substrate is plated, whereas Knapp plated on both sides. This modification minimized machining problems in that a reference surface (the unplated side of the substrate) was always available for checking dimensions.

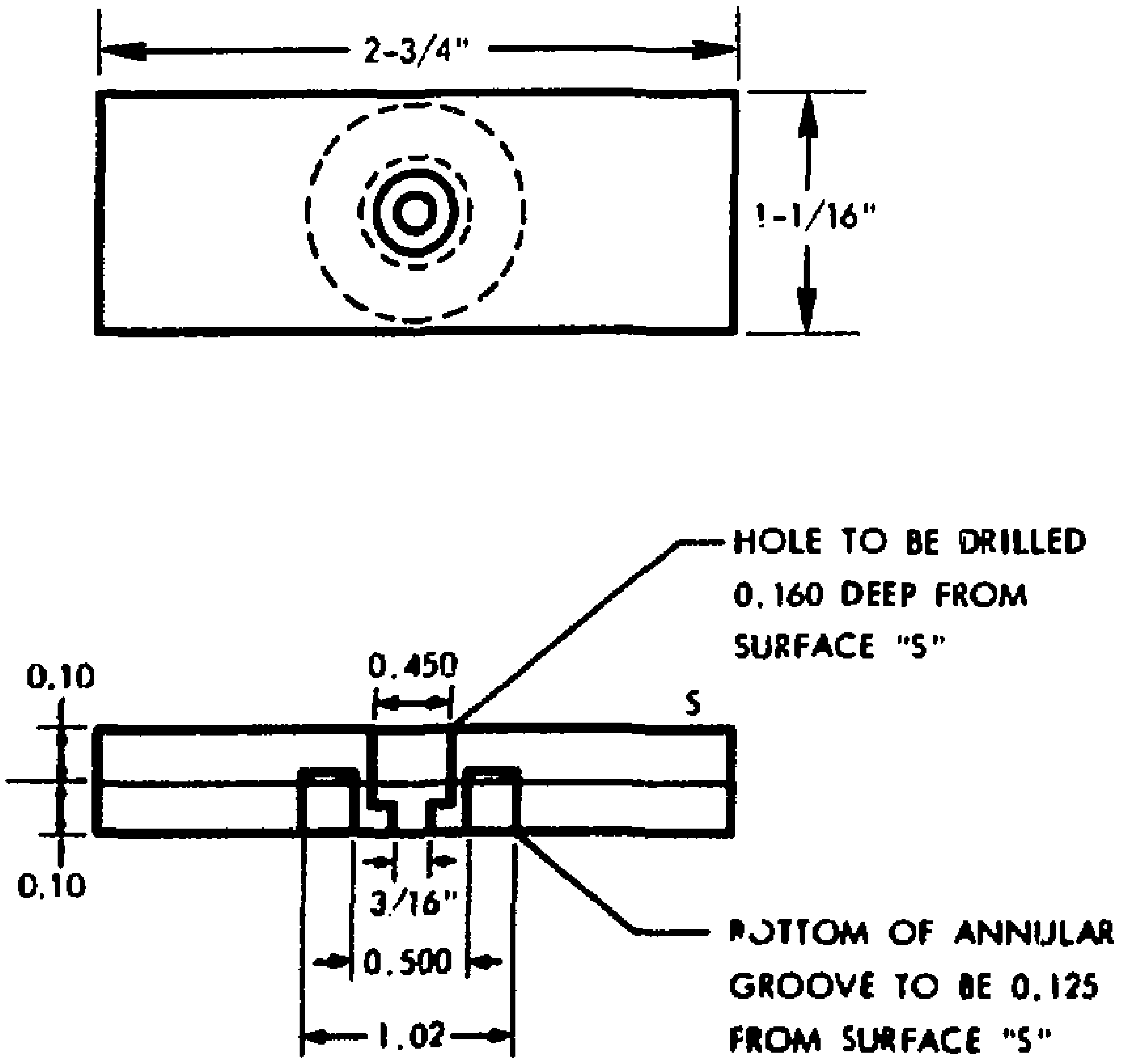


Figure 8. Cross Sectional View of Modified Ollard Test Specimen

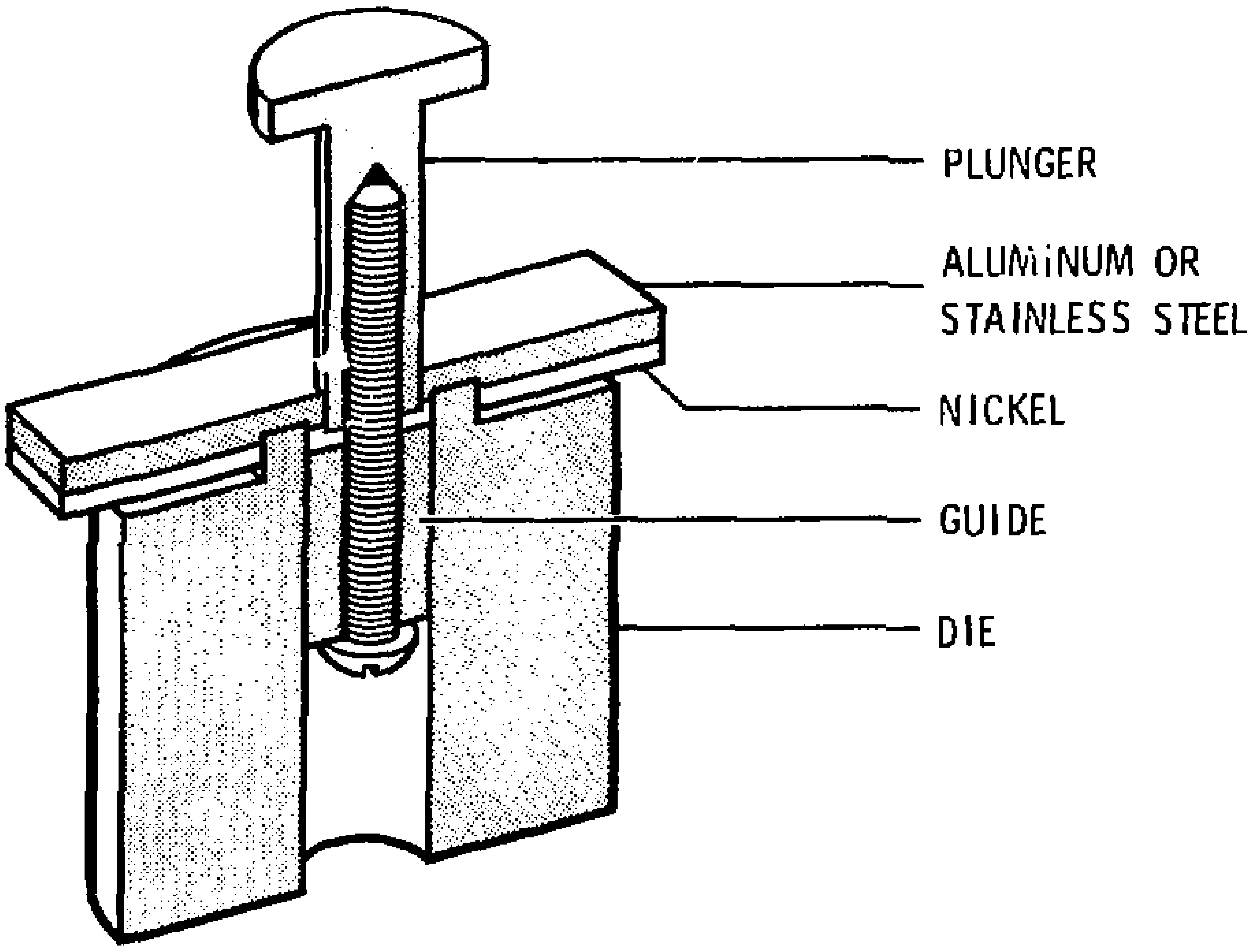


Figure 9. Modified Ollard Test Setup

Typical Applications

Modified Ollard tensile data were used to evaluate variations in the zincating cycle used prior to plating 6061-T6 aluminum with copper. The results, presented in Table V, show that all variations included in the test program provided approximately the same results. That is, failure occurred in the copper strike deposit used after zincating rather than at the interface between the plating and the aluminum. The slight reduction in adhesion shown for the double zincating operation was within the experimental error of the test.

In another series of experiments, modified Ollard test data were obtained for electroformed sulfamate nickel deposits to determine the strength of the nickel when loaded normal to the original plating substrate. In this instance, the original substrate was aluminum which was removed chemically in caustic solution before machining the test samples. Results of six tests showed that the strength of the nickel was 647 MN/m^2 (93,800 psi); these results were quite similar to values of 626 MN/m^2 (90,700 psi) obtained when the same material was tested under a loading condition parallel to the original substrate.

TABLE V

MODIFIED OLLARD TENSILE DATA FOR COPPER-PLATED 6061-T6 ALUMINUM

Treatment Cycle(A)	Ollard Tensile Strength (MN/m^2)	(psi)
Zincate(B) (30 seconds) Plus Copper Strike(C)	166	24,000
Zincate(B) (60 seconds) Plus Copper Cyanide Strike(C)	166	24,000
Double Zincate(B)(D) Plus Copper Cyanide Strike(C)	145	21,000

(A) All specimens were overplated with acid copper deposits (UBAC, Product of Udylyte Corp., Detroit, Mich.) after the copper cyanide strike.

(B) 525 g/l sodium hydroxide; 98 g/l zinc oxide; 10 g/l Rochelle salt; 1.0 g/l ferric chloride.

(C) 41 g/l copper cyanide; 49 g/l total sodium cyanide; 30 g/l sodium carbonate; 60 g/l Rochelle salt; current density was 107 A/m^2 (10 A/ft^2).

(D) The first zincate treatment was for 60 seconds; the second was for 30 seconds.

I-Beam Tests

I-beam tests are another method for tensile testing substrate-electrodeposit combinations. The advantage of this test is that many more specimens can be prepared from one plated sample than can be prepared from a sample of the same size for the other tests described in this report. Also, the cost per sample is considerably less for I-beam specimens because of the minimal amount of machining time required. Therefore, I-beam tests are ideally suited for programs requiring many quantitative tests, such as statistical evaluations of processes.

The preparation of the specimens can best be understood by the use of an example where the bond strength of cyanide copper on stainless steel was being evaluated. Parallel grooves 6.4 by 6.4mm (0.25 by 0.25 inch) deep spaced 1.57mm (0.062 inch) apart are machined on one side of a 152 x 152 x 12.7mm (6 x 6 x 1/2 inch) stainless steel plate. The stainless steel plate is then given a Wood's nickel strike followed by the cyanide copper coating. Copper plated aluminum strips, the same width and length but 0.051 to 0.075mm (0.002 to 0.003 inch) higher than the grooves in the stainless steel are forced into the grooves (Figure 10). The part is then cleaned and plated with a minimum of 2.54mm (0.100 inch of nickel) and machined (Figure 11). The plated part is then cut into the desired specimen shape, and the aluminum is dissolved. Figure 12 shows typical I-beam specimens with the special grips used for tensile testing.

Figure 10. Early Stages of Preparation of I-Beam Test Specimens

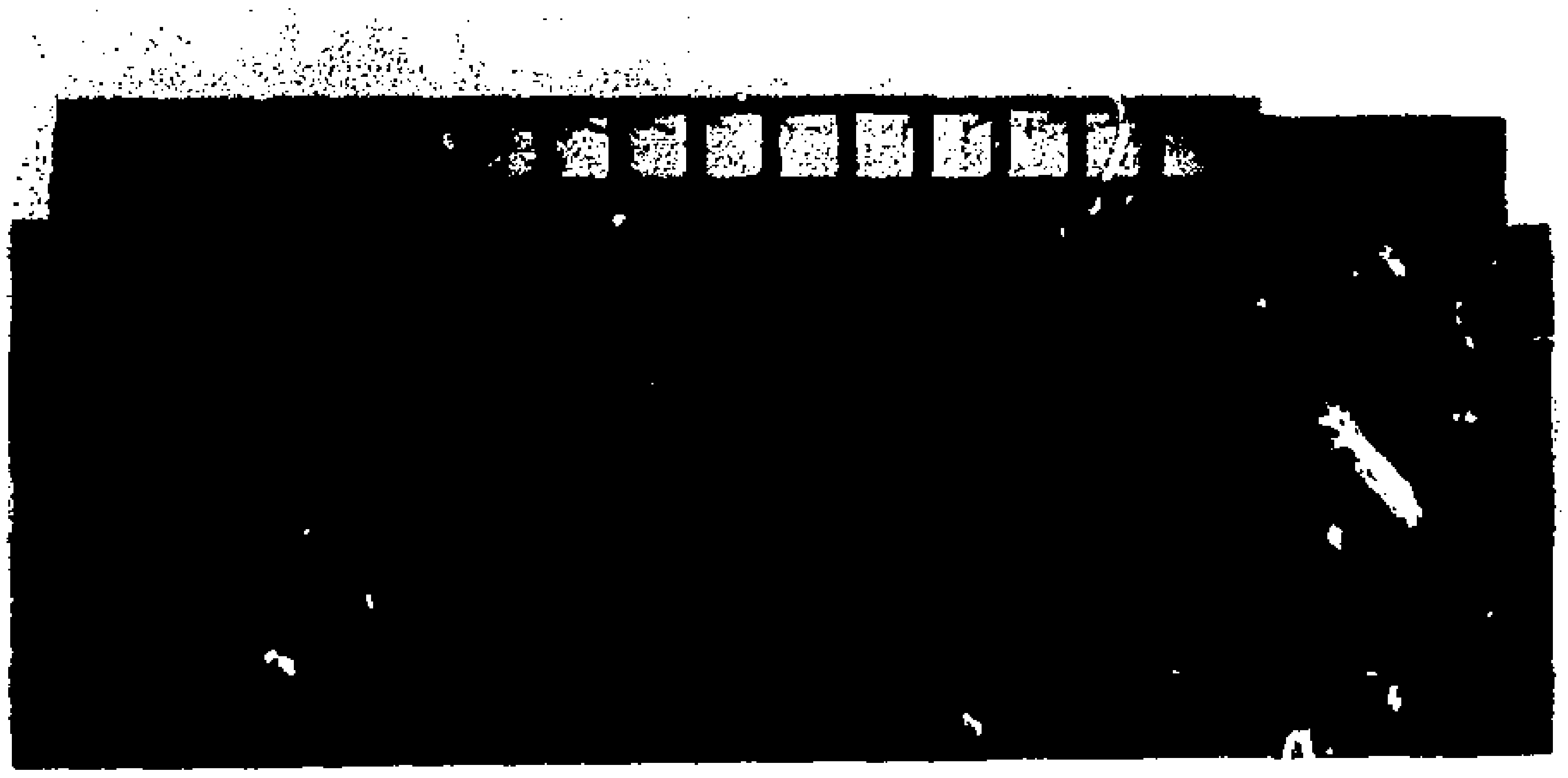


Figure 11. I-Beam Panel After Dissolution of Aluminum Strips

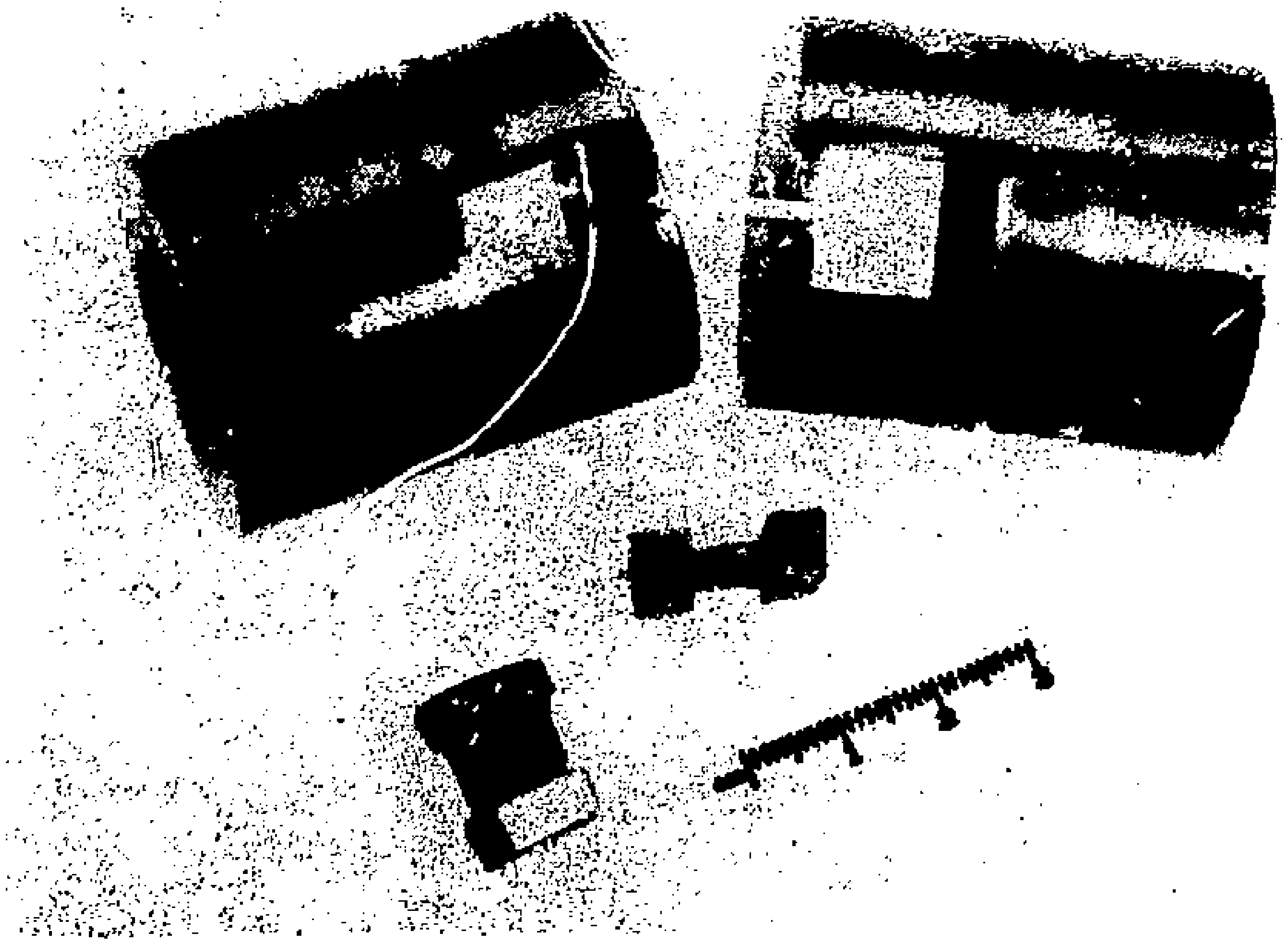


Figure 12. I-Beam Specimens and Grips Used for Testing

Typical Applications

I-beam tests were used to evaluate the influence of temperature on the bond between electrodeposited nickel and 304 stainless steel. The stainless steel was given a copper strike before nickel plating as described above.

The I-beam test data, which were obtained at room temperature, are shown in Table VI. Each value is the average of five tests, all of which were quite reproducible. The bond strength of the nickel-plated stainless steel was quite good after 30 hours at 316 C (338 MN/m^2). Some reduction was noted for specimens heated at 316 C for 100 hours; however, 251 MN/m^2 is still a quite respectable bond strength. Severe reductions in strength were noted for samples heated at 538 C. Metallographic inspection revealed that these reductions were due to Kirkendall voids formed by diffusion of a copper strike deposit into the nickel. Based on this observation, it was concluded that the strength could probably be noticeably improved by eliminating copper from electroplated parts intended for high-temperature applications.

TABLE VI

I-BEAM TEST DATA
FOR NICKEL-PLATED 304 STAINLESS STEEL

Specimen Treatment(A), (B)	Strength(C) (MN/m^2)	Strength(C) (psi)
30 hours at 316 C (600 F)	338	49,000
100 hours at 316 C	241	34,900
30 hours at 538 C (1000 F)	22	3,200
100 hours at 538 C	4	600

(A) The substrate was cleaned, subjected to a Wood's nickel strike, a 0.013-mm (0.5-mil) thick copper strike, and then plated with 2.54 mm (100 mils) of nickel from a sulfamate solution.

(B) All testing was done at room temperature.

(C) Each reported value is the average of five tests.

Flyer Plate Tests

Flyer plate tests are used for quantitatively measuring the adhesion of plated coatings under dynamic loading conditions. The method, originally developed at Sandia Laboratories, Livermore for shock wave testing materials, consists of utilizing magnetic repulsion to accelerate thin, flat, metal flyer plates against the substrates under test in a vacuum. The flyer plate test apparatus, shown in Figure 13, consists of two conductors, a ground plate, and a flyer plate, separated by a thin insulation film of plastic. The conductors are connected so that the current flowing through them produces a magnetic repulsive force that drives the thin flyer (0.2 to 1.02 mm) away from the relatively heavy ground plate and into the target specimen suspended above the flyer. The output of the flyer plate is determined by measuring the flyer velocity with a streaking camera. This velocity, coupled with metallographic cross sections of the specimens, provides the information needed to quantitatively compare different samples. Figure 14 shows a view of the flyer plate test facility.

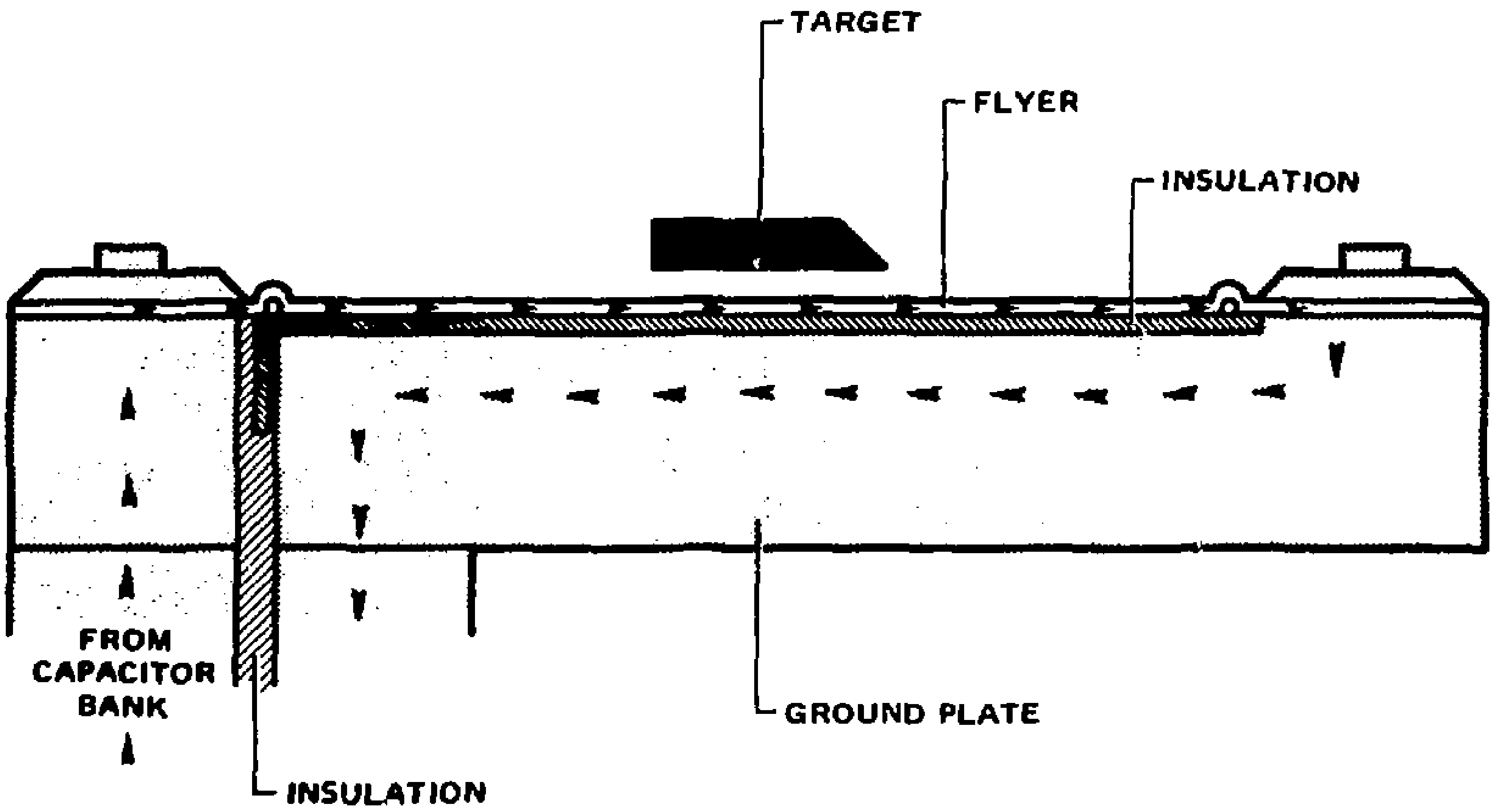


Figure 13. Flyer Plate Test Apparatus

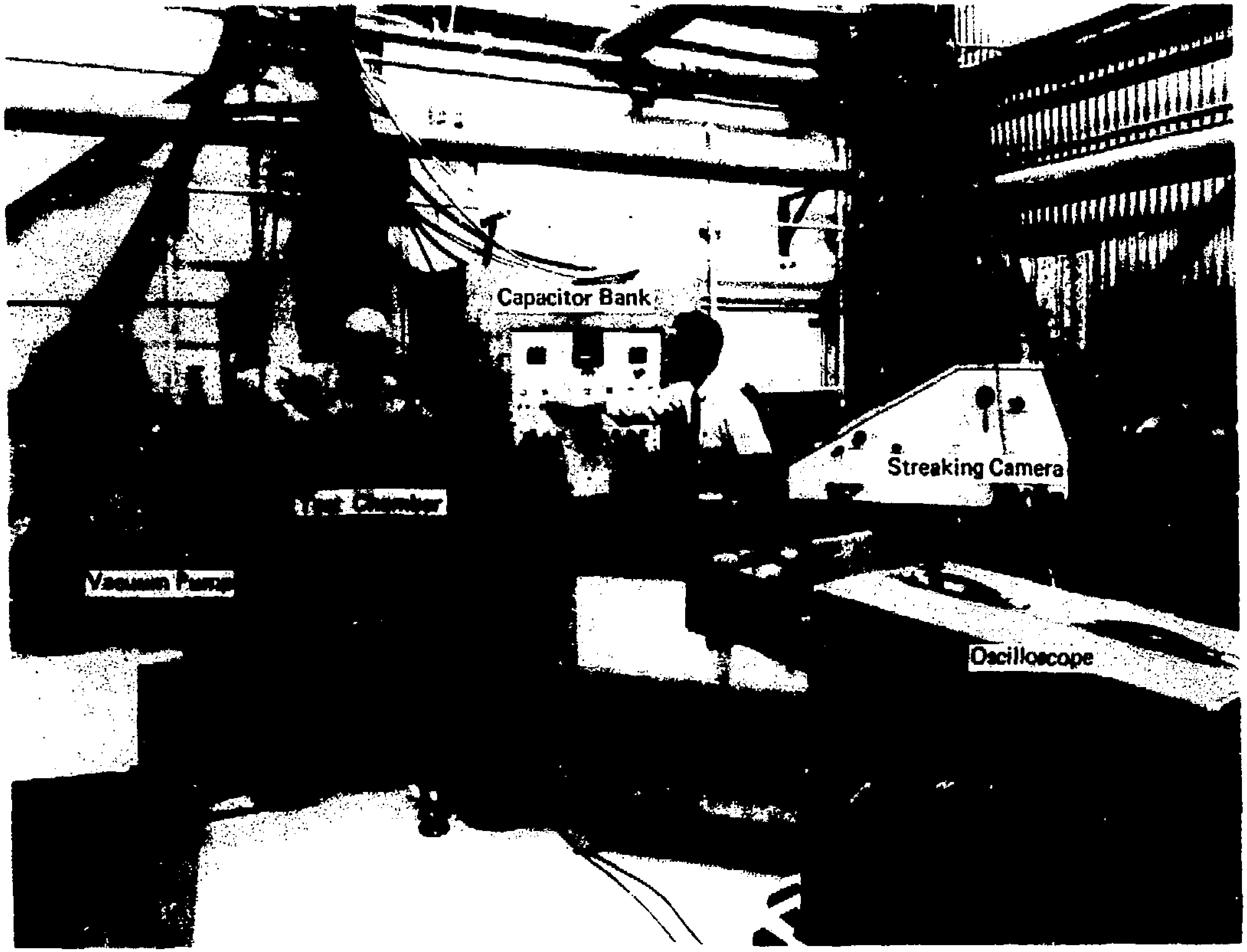


Figure 14. Flyer Plate Test Facility

When the flyer impacts the target specimen, a compressive wave is transmitted through the specimen. As it reaches the rear surface, it is reflected as a tensile wave which propagates back through the specimen. This tensile wave, combined with rarefaction waves from the front surface and waves from the impedance mismatch at the interface between the substrate and the coating, subject the interface to dynamic tensile stresses. The coating thickness and flyer plate thickness are adjusted during fabrication so that the peak tension stress will occur at the interface. Figures 15 and 16 show samples with various stages of spall.* Incipient spall flyer velocity (Figure 15) is defined as the level at which partial separation is first noted microscopically; separation spall (Figure 16) is the level at which a layer completely separates from the rear surface of the target material. Dynamic forces with amplitudes up to 100 kilobars and durations ranging from 100 to 500 ns have been attained with Sandia's flyer plate apparatus (14 kj capacitor bank).

To prepare the flyer plate test specimens, substrate plates approximately 63.5 by 127 mm (2.5 x 5 inches) are machined to a thickness of 2.54 mm (0.1 inch). One side of the panel is masked with stop-off lacquer prior to the cleaning/plating cycle. Specimens are then overplated with approximately 0.254 to 0.305 mm (10 to 12 mils) of plating, and the flyer targets 25.4 by 25.4 by 2.72 mm (1 by 1 by 0.107 inch) are machined as shown in Figure 17. The substrate thickness of 2.54 mm (0.10 inch) and plating thickness of 0.178 mm (7 mils) after machining are used because these thickness combinations used with 0.25-mm (10 mil) thick aluminum flyers produce the maximum impulse loading at the interface between the substrate and the plating for the particular materials used. For more detail on flyer plate testing, the reader is referred to Reference 14.

Typical Applications

As shown by Table VII, flyer plate tests are noticeably more discriminating than either tensile or ring shear static tests in assessing the strength of plated bonds. For example, with AM363 stainless steel, the flyer plate tests showed a significant improvement in bond strength when the acidity of the Wood's strike solution was increased by raising the HCl content of the solution from 120 to 375 ml/l. Specifically, the spall threshold for specimens treated in a solution containing 120 ml/l HCl prior to copper plating was 0.038 cm/μs; when the solution contained 375 ml/l, the spall threshold was 0.080 cm/μs. This increase indicated a better than twofold improvement in adhesion. By contrast, conical-head tensile specimens showed no difference regardless of the acidity of the Wood's strike.

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*Spall is the fracture of a material in tension caused by the interaction of two rarefaction waves.

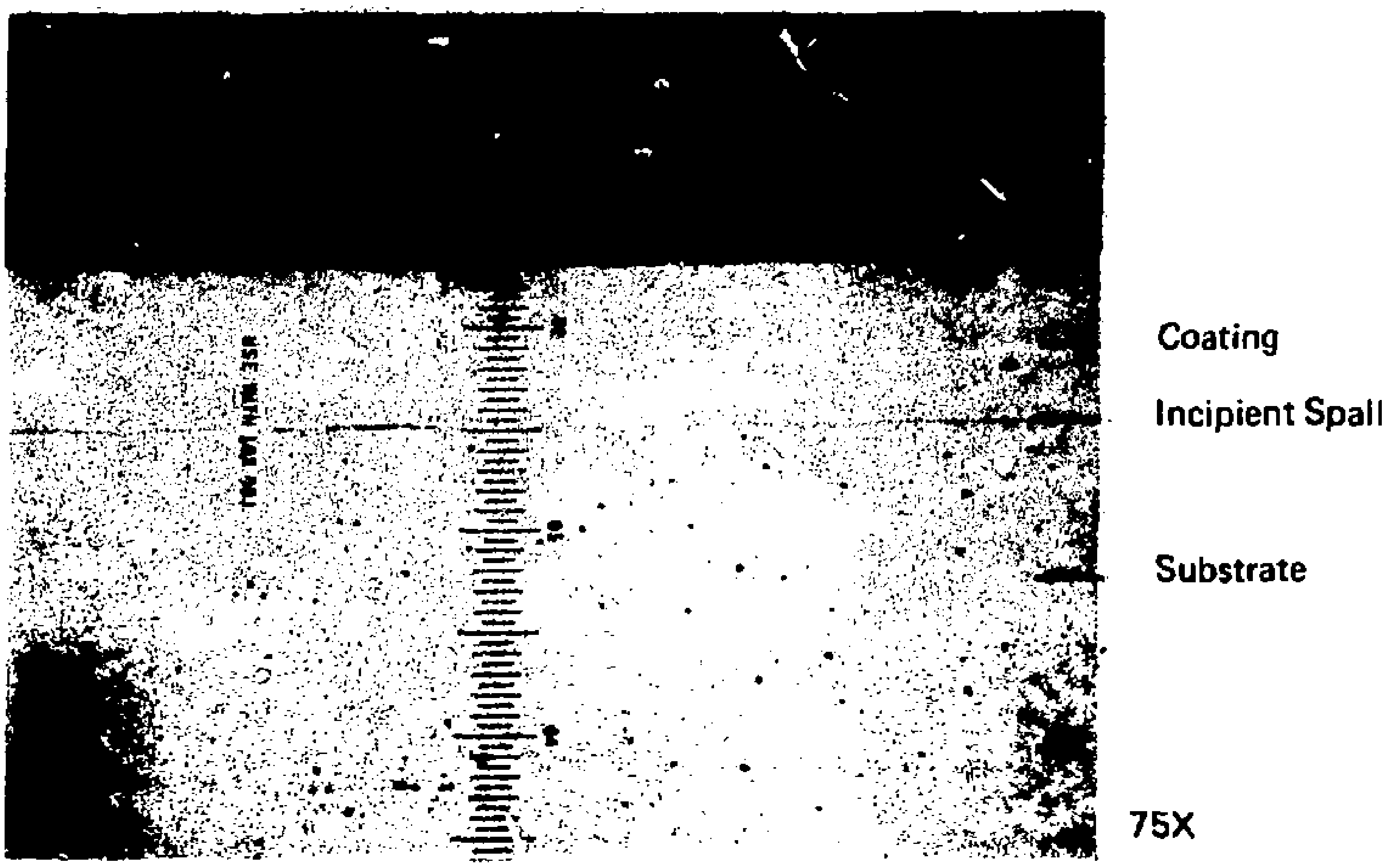


Figure 15. Example of Incipient Spall

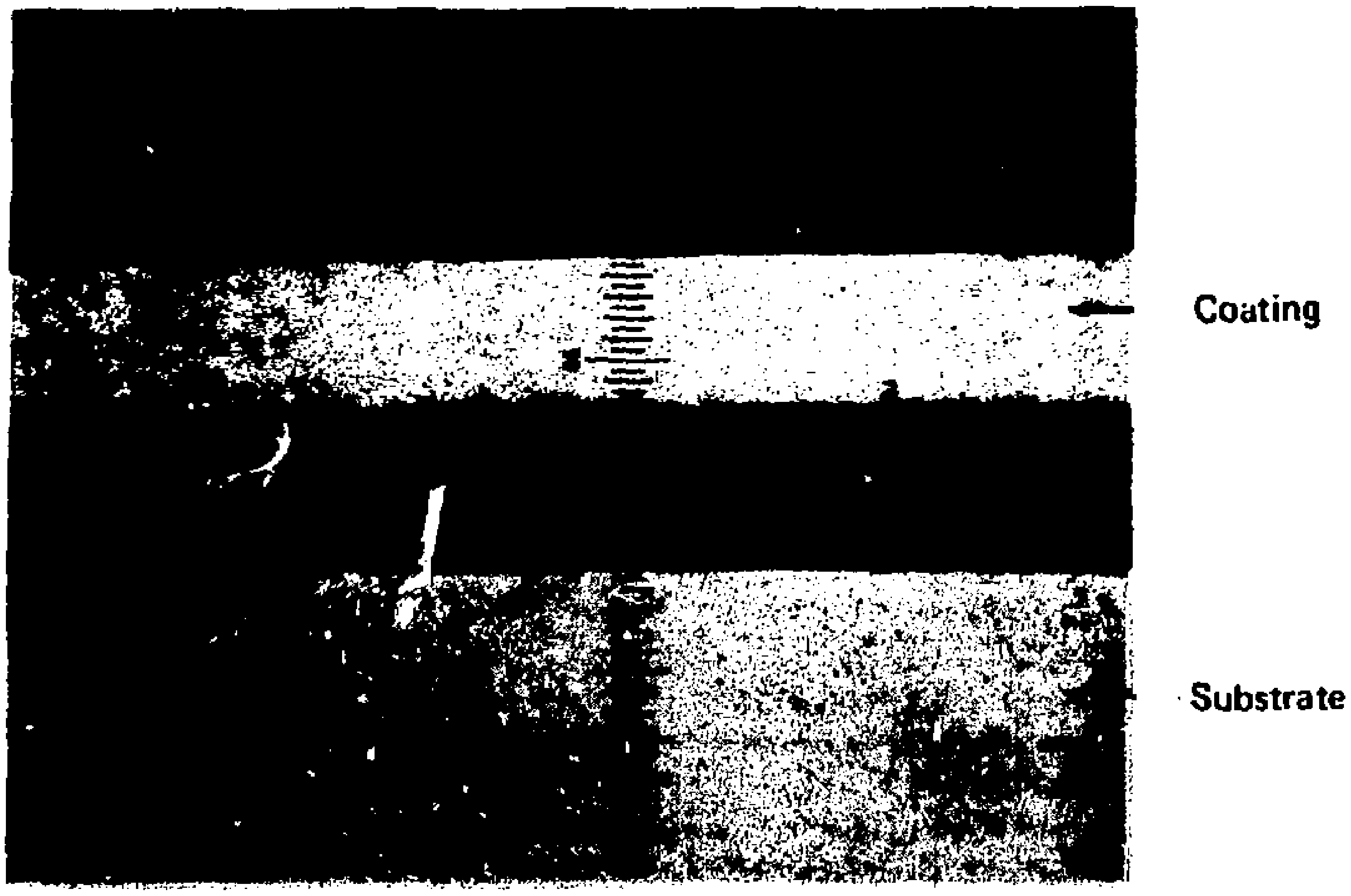
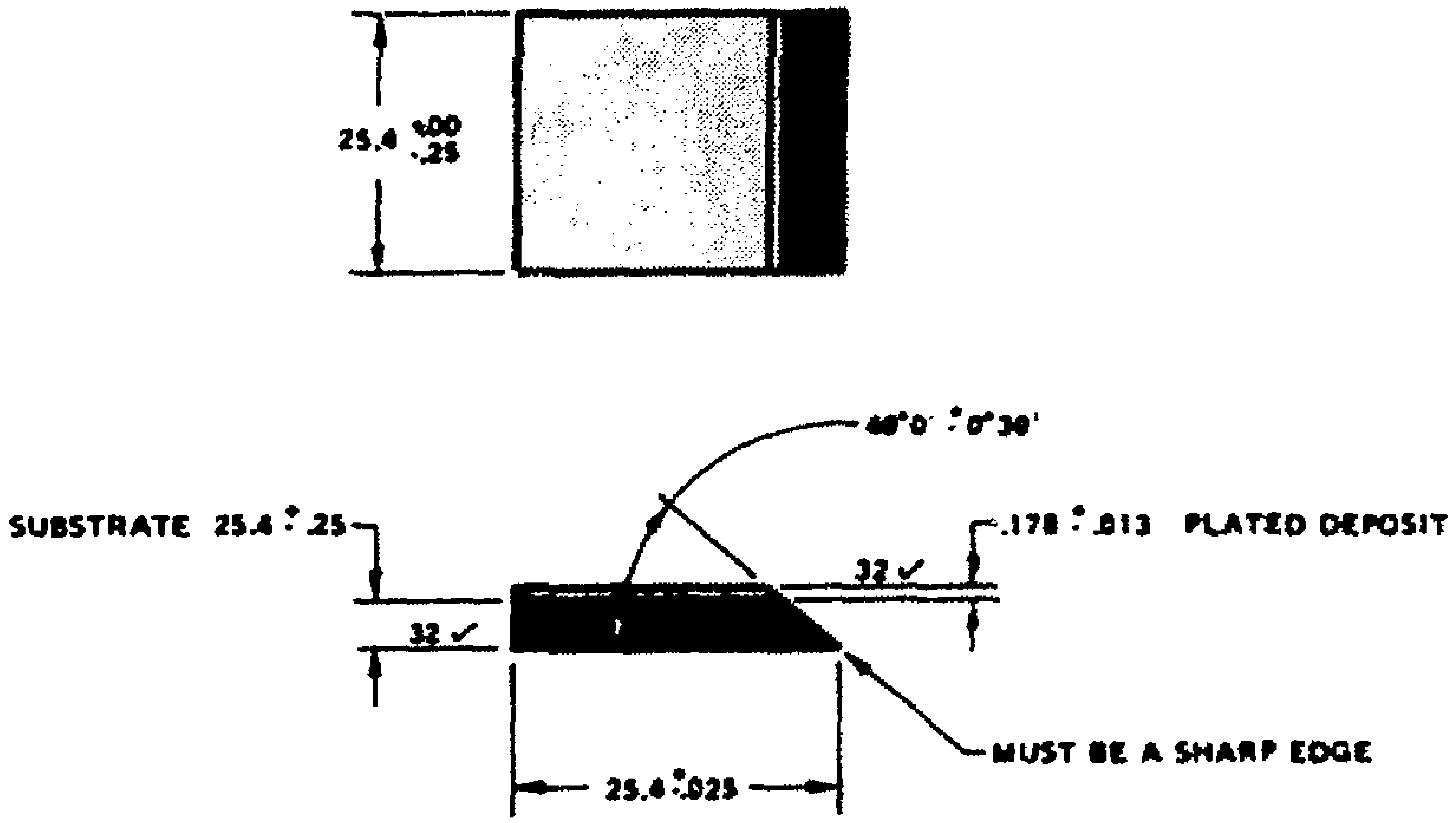


Figure 16. Example of Separation Spall



(ALL DIMENSIONS ARE IN mm)

Figure 17. Flyer Plate Target Fabrication Requirements

TABLE VII

COMPARISON OF FLYER PLATE DATA WITH STATIC TEST DATA

Substrate	Electrodeposit	Test Variable		Flyer Plate Spall Threshold Velocity (cm/μs)①	Static Tensile Strength② (MN/m²)	(psi)
		Acidity of Wood's Strike④				
AM363	Cu	$NiCl_2 \cdot 6H_2O$ HCl	240 g/l 60 ml/l	0.031	377	54,600
"	"	$NiCl_2 \cdot 6H_2O$ HCl	240 g/l 120 ml/l	0.032	352	51,000
"	"	$NiCl_2 \cdot 6H_2O$ HCl	240 g/l 175 ml/l	0.050	399	57,900

Substrate	Electrodeposit	Test Variable	Flyer Plate Spall Threshold Velocity (cm/μs)①	Ring Shear Strength③ (MN/m²)	(psi)
		Anodic H_2SO_4 Treatment Plus Wood's Strike versus Wood's Strike Alone⑤			
AM363	Ni-Co	Wood's strike	0.046	559	81,000
"	"	Anodic sulfuric plus Wood's strike	0.072	559	81,000
"	Ni	Wood's strike	0.052	455	66,000
"	"	Anodic sulfuric plus Wood's strike	0.077	455	66,000

[1] 1.0 cm/μs = 33,000 ft/s.

[2] Conical-head tensile specimens per reference 8.

[3] Ring shear specimens per reference 7.

[4] The complete preparation cycle included anodic cleaning in hot alkaline solution, rinsing, immersing in 18% (wgt) HCl at room temperature for 1 minute, rinsing, then Wood's nickel striking at 270 A/m² (25 A/ft²) for 5 minutes prior to copper plating Udylyte Bright Acid Copper (UBAC), product of Udylyte Corp., Detroit, Mich.

[5] The complete preparation cycle included anodic cleaning in hot alkaline solution, rinsing, immersing in 18% (wgt) HCl at room temperature for one minute, rinsing, anodic treating in 70% (wgt) sulfuric acid at 1080 A/m² (100 A/ft²) for 3 minutes, rinsing, and then Wood's striking at 270 A/m² (25 A/ft²) for 5 minutes prior to nickel or nickel-cobalt plating in sulfamate solution. In some cases, the anodic treatment in sulfuric acid was omitted as indicated above.

In another study with AM363, flyer plate data were compared with ring shear data. In this case, the effectiveness of utilizing an anodic treatment in sulfuric acid solution prior to a Wood's strike was evaluated. Ring shear tests showed no difference regardless of which treatment was used prior to a final plating of either nickel or nickel-cobalt. However, flyer plate tests showed a 50% improvement when the sulfuric acid anodic treatment was used prior to Wood's striking.

In both of these studies, if static tests had been the only means to quantitatively assess the bond strength, plating techniques would have been used that would not have provided maximum adhesion in a dynamic test environment. The flyer plate tests clearly provided the discrimination needed to properly assess the various treatment cycles in a quantitative fashion.

Discussion

From a practical standpoint, two important testing considerations are (1) what is the cost per test for each technique, and (2) how much time is required to prepare specimens and test them. Table VIII is presented in an attempt to answer these questions. Many metals come in "off-the-shelf" sizes that would satisfy substrate requirements for ring shear, conical-head tensile and Ollard specimens. This is not the case for either I-beam substrates, which require about three hours of machining time, or flyer plate substrates, which require about one hour of machining.

Plating thickness required for the various tests range from 10 mils for the flyer plate specimens to 200 mils for the conical-head specimens. Plating thickness requirements for the other tests range between these values. Therefore, assuming a constant plating rate, some specimens (particularly conical-head and I-beam) require longer plating times.

Final machining operations range from a simple bandsaw cut for I-beam specimens to somewhat precise machining operations for modified Ollard, conical-head, and flyer plate specimens. Final dimensions of ring shear specimens are not too critical; these specimens can be quickly machined on a lathe.

Lastly, the testing of ring shear, conical-head, modified Ollard, and I-beam specimens is straightforward, rapid, and requires only a tensile testing machine. Flyer plate tests are not as simple to perform and, in addition, require a facility costing at least $30,000.

TABLE VIII

SAMPLE PREPARATION TIME STUDY(A)

Test	Substrate Dimensions	Time to Machine Substrates	Minimum Plating Thickness (mm)	(mils)	Time to Machine One Specimen After Plating	Time to Test One Specimen
Ring Shear	12.7 mm diameter rod (1/2 in.)	0(C)	1.52	60	20 minutes	5 minutes
Conical-Head	3.2 or 6.4 mm thick plate (1/8 or 1/4 in.)	0(C)	5.10	200	2 hours	10 minutes
Modified Ollard	3.2 or 6.4 mm thick plate (1/8 or 1/4 in.)	0(C)	1.27	50	15 minutes(E)	10 minutes
I-Beam	12.7 or 19.0 thick plate by 152 mm (1/2 or 3/4 in. by 6 x 6 in.)	3 hours	2.54	100	2 minutes	5 minutes
Flyer Plate	3.2 mm thick plate by 63.5 mm by 63.5 mm by 127 mm (1/8 x 2.5 x 5.0 in.)	2 hours(D)	0.25	10	2 hours	1 hour

(A) In all cases, the assumption is made that the substrate material is stainless steel and the plating is either nickel or copper.

(B) The assumption is made that 12.7-mm (1/2-in.) diameter rods are "off-the-shelf" stock items.

(C) The assumption is made that 3.2 or 6.4-mm thick plate is "off-the-shelf" stock material.

(D) The assumption is made that the substrate material is nonmagnetic. If it is magnetic, machining time would only be one hour because grinding could be used. Time estimates include using a special fixture designed for producing the angle shown in Figure 17.

(E) Specially designed form tools are used.

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