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AN ADHESION TESTER FOR
EVAPORATED METAL FILMS

by

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ABSTRACT

A tensile tester for measuring the bond strengths of vapor-deposited metallic coatings is described. Film adherence values are read directly in pounds pull from a precision-force gage. Accuracy of the force gage is ± 0.5 percent of the full scale reading over the entire range.

SUMMARY

Bond strengths of aluminum and gold coatings have been determined on substrates of aluminum and uranium. The upper range of measurements is limited by the adhesive strength of the epoxy used to cement grip blocks to the film surface. This strength was found to be around 8000 psi.

Speed of operation is an important feature of this tester. Total film test time is limited only by the adhesive drying period because the actual test only requires seconds to perform.

INTRODUCTION

The adherence of vapor-deposited films is related to substrate surface cleanliness. Frequently it is necessary to measure this adherence in specific terms. A

Hunter-Spring* tensile tester, Model TT-150-DT has been modified to permit measurement of metal film adherence in pounds per square inch.

The commonly used Scotch tape adherence test is limited to about 75 psi which is considerably under the actual adherence of vapor-deposited coatings on clean substrates. Metal film adherence values exceeding 2000 psi have been measured with the instrument described in this report.

EXPERIMENTAL

A. DESCRIPTION

The Hunter-Spring pull tester consists of an air motor which operates in both directions, a precision force gage, holding fixtures, and a base. The variable speed air motor applies the test load, the fixtures hold the material to be tested, and the precision gage measures the load applied. Since the holding fixtures provided with the commercial instrument were designed to measure the breaking strengths of wire, a modified fixture was designed for testing adhesion of deposited films on flat substrates. Figure 1 shows the complete instrument with modification for rectangular coupons. Figure 2

* Hunter-Spring, A Division of Ametec, Inc., One Spring Avenue, Lansdale, Pennsylvania.

Figure 1. ADHESION TESTER

10070-X

shows the individual pieces of the modified fixturing for rectangular coupons. A similar device for circular coupons was also constructed.

Figure 2. MODIFICATION FIXTURE

10070-11

The air motor is equipped with an on-off control valve, manually actuated by a button; its moving piston connects directly to a set of serrated clamping jaws to grip a specimen. Opposite these jaws is a force gage which measures tension forces to 500 pounds. This gage is equipped with a "hold at maximum" indicator and a "touch" reset tab. The air motor operates forward and backward and its speed can be adjusted. An optional hydraulic check valve was obtained to provide even finer speed adjustment. A filter-regulator-lubricator was installed in the air line to protect the instrument.

The modification fixture designed for film adhesion testing consists of a set of adjustable discs, slotted to hold the specimen to the force gage. A grip block fixture mounts in the serrated clamps of the air motor (see Figures 1 and 2).

B. OPERATION

Grip blocks of aluminum are attached to the film specimen with two parts of D.E.R.®* 334 epoxy resin and one part of D.E.H.®* 12 epoxy hardener. Both are liquid at room temperature. An equally effective mixture is that of two parts of D.E.R. 332 LC and one part of D.E.H. 12. The D.E.R. 332 LC resin is a solid at room temperature but becomes liquid at 55°C. It can then be mixed with the hardener and will retain its liquid state for several hours. Curing time was twenty-four hours at room temperature or one hour at 140°C. Epoxy resin based on the reaction of bisphenol-A and epichlorohydrin is chemically inert during conversion to a thermoset solid, so are unreactive with common metal.

* Trademark of The Dow Chemical Company.

When the test specimen is in position and the air motor control button is pressed, the jaws automatically close around the grip block holder, and the piston moves at a preset speed. The piston moves at a steady rate and pulls the jaws until the adhesive bond breaks or the film is pulled away from the substrate. The indicator hand on the force gage dial holds the maximum reading to show the force exerted at the instant of failure.

As soon as breakage occurs, the operator releases the control button, the cylinder returns to its starting position and the jaws open. After the reading is noted, the indicator is reset to zero by touching the release tab. Air motor ram speed can be controlled down to six inches per minute, with lower rates of travel provided by the hydraulic check valve. A speed of four inches per minute was found to be most effective.

DISCUSSION

Initial tests were made to determine the bond strength of the epoxy cement. D.E.R. 332 LC and D.E.H. 12 in a two to one mixture was evaluated with a 500-pound force gage. Grip blocks of aluminum were cemented to aluminum coupons without a deposited film. Curing time was one hour at 140°C. Table I shows the pressures reached before the adhesive failed. This

TABLE I

BOND STRENGTH OF EPOXY MIXTURE
OF D.E.R. 332 LC AND D.E.H. 12 ON
UNCOATED ALUMINUM SUBSTRATE

Force Gage Reading (lbs.)	PSI to Break Bond
297	7425
320	8000
320	8000
200	5000
345	8625
325	8125
305	7625
340	8500
310	7750
310	7750

data indicates some measure of reproducibility of the method. The fourth test in the group showed a considerably lower yield point. The grip block, in this instance, had moved a quarter of an inch during curing.

Using identical coupons and grip blocks, adhesion tests were made on evaporated films of aluminum and gold as designated in Table II. The thickness of the films averaged 3500 angstroms $\pm 30\%$. Aluminum and

TABLE II

BOND STRENGTHS OF EVAPORATED FILMS

Al Film on U Substrate (psi)	Au Film on U Substrate (psi)	Au Film on Al Substrate (psi)	Al-Au Film on U Substrate (psi)
1975	850	3200	1875
2625	1400	1800	1750
2725	900	2450	1800
2525	1275	2525	1700
2350	1250	1875	
2125	1200	2150	
2450		2750	

uranium substrates were in the form of circular discs. The aluminum was hand polished and the uranium electropolished prior to vacuum coating. Epoxy mixture.

of D.E.R. 332 LC and D.E.H. 12 was used with a curing time of one hour at 140°C.

The evaporated films could not be removed by Scotch tape in any of the tests in Table II. In the test of the Al-Au sandwich film, separation in each case was at the uranium substrate. The first film was a flash coating of aluminum in the order of 500 angstroms, followed by a gold film of approximately 3000 angstroms.

Visual observation of the removed films on the ends of the grip blocks indicated a clean removal with no epoxy penetrating into the substrate. In order to test thick films (greater than 10,000 angstroms), it would be advisable to have a substrate surface the same size as the grip block.

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