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Copper Metallization of Stainless Steels

Kansas City Division

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KCP-613-5435

Published September 1994

Final Report

Approved for public release; distribution is unlimited.

Prepared Under Contract Number DE-ACO4-76-DP00613 for the
United States Department of Energy

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A prime contractor with the United States
Department of Energy under Contract Number
DE-ACO4-76-DP00613.

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KCP-613-5435
Distribution Category UC-706

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Technical Communications
Kansas City Division

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Abstract

Stainless steel plates were vacuum metallized with pure copper films 0.004 inch thick. Prior to metallization the surfaces were cleaned with EPA-approved detergents. A thin layer of chromium was sputtered for improved adhesion followed by sputtering of the copper. Results showed excellent adhesion of the sputtered films to the substrates.

Summary

Modern technology requires the application of thin solid films on a variety of substrates. The most prevalent failure noticed in these systems is poor, or lack of, adhesion between the film and the substrate. This results in the exfoliation of the film, leading to failure. Poor adhesion, in most cases, is attributed to surface contamination from improper substrate preparation or handling following the preparation. There are numerous techniques for substrate preparation depending on the substrate, but all stress the mandatory removal of all types of contaminants and particulates from the surface before metallization.

excellent adhesion between the copper and the substrate.

This study demonstrated that clean metallic surfaces can be obtained using water-soluble detergents.

In this study, a thorough surface preparation was conducted by the use of water-soluble detergents approved by the EPA. Cleaning and handling procedures were developed and tested to ensure absolutely clean surfaces of the substrates prior to metallization. Stainless steel plates were metallized with high purity copper, with a thin layer of pure chromium as the adhesion layer, by vacuum sputtering. The results showed

Discussion

Scope and Purpose

This project was initiated to develop procedures for vacuum metallization, by sputtering, of pure copper films on stainless steel substrates. This process would replace electroplating because it generates no hazardous waste and is considered environmentally friendly. The main thrust of this study was the demand for excellent adhesion of the sputtered film to the substrate. The objective was to develop procedures and materials approved by the EPA to be environmentally safe, and to implement the procedures as a replacement for electroplating.

Several water-soluble detergents and plasma cleaning procedures were evaluated for surface preparation and their influence on adhesion and physical properties of the sputtered films.

Activity

Thin films of high purity copper were vacuum sputtered on commercially available stainless steel substrates. Vacuum sputtering is a process by which thin solid films are deposited on solid surfaces of all sizes and shapes. Substrate preparation is critical to the mechanical and physical properties of the films. In most cases the dominant failure mechanism, attributable to surface contamination, is lack of adhesion between the film and the substrate.

In this study, several surface treatment procedures were evaluated, such as plasma cleaning and degreasing with water-soluble, commercially available detergents. Three dishwasher detergents were evaluated for their ability to degrease metallic surfaces.

Plasma cleaning is known to produce clean surfaces suitable for vacuum metallization. This process was used as a standard for comparison to other surface cleaning processes with water-soluble detergents. Surfaces were characterized by advanced electron optical techniques such as Auger electron spectroscopy (AES) and X-ray dispersion.

Samples were cleaned, vacuum metallized, and tested for adhesion by subjecting them to uniaxial tension.

The as-received stainless was sectioned to 1- by 1-inch coupons, and they were degreased using d-Limonene in an ultrasonic cleaner, followed by thorough rinsing with hot DI water and drying in an oven at 120°C. Analyses of d-Limonene cleaned surfaces pointed out a residual thin organic on the surfaces. A thorough cleaning with dishwasher detergent and rinse with hot DI water satisfactorily removed the residual films. The samples were then placed in the vacuum sputtering chamber and heated in vacuum (10^{-6} torr) to 120-150°C. After metallization in an argon atmosphere of

10⁻³ torr, a thin layer (200-500 angstroms thick) of chromium was sputter deposited to act as an adhesion layer; and then, without breaking vacuum, high-purity copper was sputtered to the desired thickness, followed by slow cooling in vacuum to room temperature.

Adhesion tests were conducted by subjecting the films to uniaxial tension in an Instron tensile tester. Support rods were attached to the substrate and the copper film by epoxy resin and inserted in the Instron. Data from the pull test (Table 1) showed that the best adhesion, higher than 1500 psi, was achieved with samples cleaned in plasma and then in detergent A. In these cases failure occurred at the epoxy-copper interfaces. Samples cleaned with the other detergents exhibited lower adhesion strengths, ranging from 300 to 1000 psi.

Accomplishments

This study illustrates that surfaces can be properly cleaned with commercially available water-soluble detergents. Improved adhesion of thin films to the substrates was realized with clean surfaces.

Future Work

As the list of banned cleaner grows longer, there is an urgent need to identify new environmentally safe cleaners. Atomically clean surfaces are mandatory for all types of surface finish, and suitable cleaners should be developed and tested, especially in vacuum metallization which will replace some electroplating processes.

Table. 1. Adhesion Determined by Uniaxial Pull Test

Cleaning Agents	Adhesion (psi)	Interfacial Failure
Plasma	800 - 1200	Substrate/Chrome
Plasma/A/DI	>1500	Epoxy/Copper
A/DI	>1500	Epoxy/Copper
Plasma/B/DI	1000 - 1200	Substrate/Chrome
B/DI	1000 - 1200	Substrate/Chrome
Plasma/C/DI	300 - 600	Substrate/Chrome
C/DI	<400	Substrate/Chrome