CHAPTER-II

Literature Survey
&
Scope of the present
Investigation

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LITERATURE SURVEY & SCOPE OF THE PRESENT INVESTIGATION

2.1. Literature Survey

2.1.1. Cyanide bath

An electrochemical and spectroelectrochemical study of the electrodeposition of Au from $KAu(CN)_2$ solutions containing 4-cyanopyridine and cetylpyridinium chloride were studied by Benedetto Bozzini FIMF et al [1].

The electrodeposition of gold-copper-cadmium alloys was proposed allowing the plating of thick layers of gold-alloys with sound mechanical properties, which could not be achieved with free-cyanide gold-copper baths by Benedetto Bozzini et al [2].

Kinetics of gold electrodeposition in the presence of catalytically active thallium (I) ions while monitoring the coverage of the gold surface by thallium adatoms were decribed by R.Yu.Bek [3].

Electrokinetic behavior of gold alloy and composite plating baths were studied by B.Bozzini et al [4].

B.Bozzini et al [5] have studied the spectroelectrochemical behaviour of CN^- on a Au electrode in a $KAu(CN)_2$ bath at pH 6.3 by in-situ confocal Raman spectroscopy .

A bath for the electrodeposition of white gold alloys of interest for the electroforming of hollow jewellery is proposed by B.Bozzini. et al [6]. The investigated system was an alkaline $KAu(CN)_2$ bath for the electrodeposition of Au-Sn.

R.Yu.Bek [7] studied the kinetics of anodic dissolution of gold in cyanide solutions containing thallium ions using potentiostatic measurements and explained the possible mechanism of the acceleration of gold dissolution in the presence of thallium adatoms.

Hydrodynamic effects in the electrodeposition of Au-Cu-Cd alloys were studied by B.Bozzini et al [8]. Compositional maps of the deposits grown in the flow-cell were measured by energy dispersive spectroscopy and compared with computed fluid flow distributions.

The electrodeposition of Au-Cu-(B_4C) matrix composites from alkaline baths containing free cyanide is described by B.Bozzini et al [9]. The electrochemical behaviour of the bath and the metallographic and crystalline structures of the electrodeposited alloys were studied with and without addition of particles.

The influence of alkali metal cations on the capacitance of the electrical double layer is studied at the gold electrode in solutions containing both indifferent anions and specifically adsorbed CN^- ions by N.A. Rogozhnikov et al [10].

The gold dissolution rate in alkali cyanide solutions containing with and without bismuth hydroxyl compounds is studied as a function of potential E, with the electrode surface renewed before taking measurements [11]. Kinetics of anodic dissolution of gold in cyanide solutions containing the additives were reported [12].

The influences of temperature on the anodic dissolution of gold in cyanide solutions were studied [13]. It is shown that a temperature increase has a complex effect on the anodic dissolution of gold.

R.M. Krishnan et al [14] have reported the electrodeposition of Gold-Nickel alloys, the preparation of gold potassium cyanide from gold metal and production of gold –nickel alloy deposits of different compositions by the addition of nickel EDTA complex.

Stress was determination in pulse electroplated gold deposits from a cyanide type bath by D.L.Rehrig et al [15]. It was found that shifts in orientation and grain size accompanied the differences in measured stress.

The electrodeposition of Au-Cu-Cd alloys from cyanide baths was investigated under different hydrodynamic conditions. Alloys obtained at different current densities were characterized from the compositional, structural and morphological points of view [16].

The individual and simultaneous deposition of silver and gold from low cyanide concentration solutions showed two regions in the potentiodynamic j-E curves [17]. At low overpotentials, these processes take place through adsorbed species while at high overpotentials the normal diffusional region is observed.

The electrochemical and structural aspects of gold deposition from dilute cyanide, citrate and phosphate-buffered plating solutions were studied using rotating disk electrode [18].

Comparison of nanometer-scale structures electrodeposited on Au and Pt seed electrode using pulse/DC electroplating were studied by Y.Lee et al [19]. In addition, the effects of pulse plating variables were investigated with respect to DC plating. The results obtained from the electroplated Au films on the Au seed layer are compared with those on the Pt one.

Measurement of the mechanical properties of electroplated gold thin films using micromachined beam structures studied by Chang-Wook Beak et al [20].

L.Guzman et al [21] have studied the ion beam-induced enhanced adhesion of gold films deposited on glass. The substrates were pretreated by an ion-beam-mixing step, consisting of the deposition of Au/C bilayers or C/Au/C multilayers followed by Xe^+ implantation.

Electroplating of gold on sputtered gold microelectrode sites was studied as a means to provide a fuzzy, roughened surface for electrodeposited conducting polymers to adhere. It was found that concentration of $Au(CN)_2^-$ solution and current density played important roles in controlling the morphology of the deposits as studied by Xinyan Cui et al [22].

2.1.2. Non-cyanide baths

Eletrodeposition of gold from a thiosulfate-sulfite bath for microelectronic applications were studied by several investigators [23-30].

T.A.Green et al [31] has studied the electrodeposition of gold from a thiosulfate-sulfite bath for electronic applications. The thickness, uniformity, roughness, stress, hardness, adhesion and shape of the plated structures were found to be compatible with the requirements for a wide range of microelectronics applications, including wafer bumping.

The electrochemical reduction of gold thiosulfate were studied by Anne M.Sullivan [32], gold-thiosulfate is a potential replacement for gold cyanide in

electro and electroless plating baths. Gold thiosulfate has more positive reduction potential than gold cyanide and eliminates the use of cyanide.

Osaka and co-workers [33, 34] proposed the use of a gold plating bath containing sulfite and thiosulfate as ligands. Since the stability constant of the $Au(S_2O_3)_2^{3-}$ complex is much larger than for $Au(SO_3)_2^{3-}$ ($pKa=10^{26}$ Vs 10^{10}) the former species predominates in effect which is confirmed from the results with a gold thiosulfate bath [35].

The hardness of the soft gold electrodeposited from the thiosulfate-sulfite mixed ligand bath that was developed recently and studied by T.Osaka [34]. The mechanism of the sulfur inclusion was investigated by analyzing for sulfur in gold deposits produced under various experimental conditions and also by observing weight gains resulting from the adsorption of sulfur-containing species on gold using the quartz crystal microbalance (QCM) technique.

Fabrications of gold bumps (20 ~ 100m in size) using gold sulfite plating were studied by H.Homma et al [36]. To improve the stability of the gold sulfite plating bath, the behaviour of the decomposition procedures and the evaluation of the stabilizer were also studied.

The anodic corrosion of gold in concentrated chloride solutions as a function of chloride concentration and pH was studied by R.P. Frankenthal et al [37].

The scanning electrochemical microscope was applied in the feedback mode to deposit Au microstructures on n-Si (111) and indium-tin oxide have been reported by Erich Ammann [38].

Jean Horkans et al [39] have studied the kinetics of gold deposition from Au (I) sulfite solutions in the pH range 8.5-13 using cyclic voltammetry and single potential step methods.

Formation of gold microbump by electrodeposition using gold sulfite as a metal source was investigated by Hideto Watanabe [40] et al. gold bumps 20-80μm wide and 20μm high were fabricated by either direct or pulse current plating. Soft and uniform gold bumps can be obtained by adding 1-10 ppm of cerium ions in the plating bath.

Xiaoping Wang [41] et al have studied the electrodeposition of gold from a new electroplating system for gold that contains iodide and thiosulfate has been studied using rotating disk voltammetry. The cathodic electron transfer is slow, with a transfer coefficient of α = 0.76 ±0.02, and is coupled with a preceding chemical reaction, the formation of the complex ion Au $(S_2O_3)_2^{3-}$.

A sulfite gold plating solution is capable of stable operation at pH values as low as 4.0. At pH values lower than 6.5, sulfur dioxide is released at a controlled rate during operation. The effect of several such brightener systems on deposit grain size, crystal orientation, leveling and porosity Vs thicknesses were studied by Ronald J.Morrissey [42].

Au(I)- thiourea aqueous baths are a promising alternative to cyanide solutions for the electrodeposition of gold and its alloy as reported by Ameriga Fanigliulo et al [43]. This study is based on cyclic voltammmetry and in situ surface-enhanced Raman spectroscopy. Potential-dependent changes of orientation with thiourea additive have been recognized.

Gold electrodeposition was studied on glassy carbon and platinum electrodes from Au (I) ammoniacal solution. The current transients obtained from potential step perturbations showed a typical metallic nucleation growth controlled by diffusion studied by G.Trejo et al [44].

Gold bumps of high-performance gold plating for microdevices from a sulfitic gold electrolyte have been studied by A.Gemmler et al [45]. Sources of nodules and pits, or voids were characterized using SEM and acousto-microscopy. Further factors that influence quality of gold deposits could be optimized.

The addition of sodium laurylsulfate, decylamine and dodecylamine to the cyanide gold plating bath was found to suppress the growth of the (111) facet as studied by Kwang-Lung Lin et al [46].

The anodic dissolution of gold in alkaline thiosulfate solutions has been studied by using a rotating gold disc by Suchun zhang et al [47]. Experimental results have shown that the gold dissolves at measurable rates in thiosulfate solutions at potentials above 0.2V.

Zhanhua Wang et al [48] studied the effect of thickness and types of gold plating on the resistance to high temperature discoloration of the gold plating cavity surface of ceramic package. Non-cyanide gold plating showed better resistance to high –temperature aging than cyanide gold plating.

The corrosion of Au-Pd-In alloy, which is of great importance in industry, has been studied using an electrochemical quartz crystal microbalance in simulated physiological solutions by K.Leinartas [49].

Gold wire bondability of electroless gold plating using disulfiteaurate complex were studied by H.Watanabe [50].

Deposition of gold on copper from acid gold bath was investigated using electrochemical and microscopic techniques. The growth morphology of both soft and hard gold deposits was characterized using scanning electron microscopy (SEM) and atomic force microscopies (AFM) by Y.G.Li, A.Lasia [51].

T.Mitsui et al [52] have studied by novel preparation of gold films by electrodeposition at the interface between a Na(AuCl4)-CuSO4 aqueous phase and an immiscible hexyl acetate organic phase resulted in continuous growth of gold films along the interface.

Minute amounts of metallic as well as non-metallic inclusions that scientifically affect both bulk and surface properties of the electrodeposited gold films being used by the electronics industry were studied by Yutaka Okinaka [53].

The electroplating of gold-tin alloys from sulfite-citrate baths were studied by Y.Zhang et al [54]. The effects of bath composition and pulse current (PC) plating factors on the composition, microstructure and plating Au-Sn deposits have been investigated.

Masaru Kato et al studied [55] the plated gold being used by the electronics industry can be broadly classified into two categories: soft gold and hard gold. Soft gold is used for circuit metallization and for bonding semiconductors chips, while hard gold is indispensable as the contact materials on electrical connectors, electromechanical relays and printed circuit boards.

Electrochemical dissolution of calaverite ($AuTe_2$) in thiourea acidic solutions indicate that for potentials of about 0.4 V vs SCE adsorbed thiourea

decomposes to formamidine disulphide, while gold from calaverite oxidizes and complexes with thiourea as studied by S. Aguayo Salinas et al [56].

The electrochemical preparation of highly dispersed gold microparticles on the surface of glassy carbon (GC) electrodes and their electrocatalytic activities for the oxidation of formaldehyde were studied by H.Yang et al [57]. It was found that the reduction of Au^{3+} to Au is controlled by diffusion and the formation mechanism of Au microparticles on the GC surfaces corresponds to an instantaneous nucleation and diffusion-controlled three dimensional growth process.

The electrochemical quartz crystal microbalances as a sensor in the gold thiosulfate leaching process were studied by P.L.Breuer [58].

Non-cyanide type gold –tin alloy plating bath were studied by Uchida [59], a gold-tin alloy plating film having good brightness, reflow properties formed.

A relatively stable, weakly acidic, non-cyanide electroplating solution for deposition of Au-Sn alloys over a range of compounds were studied by Ivey at al [60]. The solution consists of Au and Sn chloride salts as well as ammonium citrate as a buffering agent and sodium sulphite and L-ascorbic acid as stabilizers.

Kitada et al [61] have been studied the process for producing bis (1,2-ethanediamine) gold chloride and gold-plating solution containing the gold chloride.

Non-cyanide electroplating solution for gold or alloys thereof; said solutions comprising gold in the form of soluble sulfite complex added source

of sulfite and/or bisulfite ion and a supporting electrolyte were studied by Morrisey et al [62].

Depositions of gold nanoparticles on silica spheres by electroless metal plating technique studied Yoshio Kobayashi et al [63]. TEM, XRD, and UV – vis absorption spectroscopy revealed that gold metal nanoparticles with an average particle size of 1.5 nm and a crystal size of 5.1 nm were formed on the silica spheres with a size of 273 nm and at an Au concentration of 0.77 M.

Nucleation and growth mechanism of sequential electroplating of gold over thick tin layer have been studied by SEM, EDX and X-Ray diffraction method [64]. The growth mechanism of Au layer on top of Sn layer is diffusion controlled process and the primal Au-Sn intermetallic compound and Au phases are $AuSn_4(211)$ and Au(111) respectively.

Development of an electroplating solution containing weakly acidic non-cyanide bath for codepositing Au-Sn alloys over a range of compositions were studied by W.Sun et al [65]. Electrochemical studies have been conducted to examine the effects of the various additives and their concentrations on the bath stability and plating bahaviour.

J.Doesburg et al [66] have studied the microstructure and preferred orientation of Au-Sn alloy plated deposits. At low current densities, the growth of existing grains is favored, while at high current densities the formation of new grains is favored and the non-cyanide electroplating solution has been used.

Microstructural characterizations of pulsed electrodeposited Au/Sn alloy thin films were studied by B.Djurfors et al [67]. The multilayer structure as a viable method of producing eutectic Au/Sn alloys for solder applications.

Electrodeposition of soft gold is an important process in the fabrication of micro devices for electronics, optics etc and characterizing of a thiosulphate-sulfite gold electrodeposition process were studied by M.J-Liew et al [68]. Standard rotating and cyclic voltammety technique have been carried out to determine the electrochemical behaviour of the gold-thiosulphate-sulfite system.

A new acidic electroplating solution has been developed for depositing of Au–Co alloys over a wide range of current densities (5–60 A/dm^{-2}). The solution consisted of Au cyanide and Co salt as well as oxalate–citrate buffer, brightener and an organic additive. This has been reported by L. Chalumeau [69].

O. Yevtushenko et al [70] reported the nanocrystalline gold is electrodeposited from non-cyanide non-sulfite bath by pulse techniques. The thermal stability of nanocrystalline gold is investigated in situ by high-temperature X-ray diffraction.

2.2. Scope of the present work

Gold is one of the most noble metals with very good electrical properties, which has made it to be a logical choice for electronic interconnection applications. Gold is applied to the surfaces of electric components by electroplating, thermal decomposition of screen-printed paste, vaccum deposition or thermal decomposition of a metal organic compound or by mechanical rolling of inlay material. The electroplated gold being used in the electronics industry can be broadly classified into soft gold and hard gold. Soft gold is used as a surface finish for bonding gold or aluminum wire in the

conventional method of mounting semiconductor devices on circuit boards. Hard gold is used as electrical connectors and contacts requiring resistance to mechanical wear as well as low electrical contact resistance.

The present research work is related to two aspects of gold plating. The first part of the work is to deal with the development of gold plating using different stabilizing agents. This type of plating was aimed in order to obtain superior functional performances over the traditional gold electrodeposition. The plating parameters affecting the formation of gold coatings using EDTA and citric acid from sulfite-thiosluphate baths have to be examined.

The second part of the work is to study the influence of some organic additives for the deposition of gold from acid non-cyanide bath. The optimum concentration for the gold electrodeposition in terms of the additive concentration and other operating parameters are to be fixed.

The choice of the electrolyte, stabilizing agents, plating bath additives, substrates and the techniques planned for studying the gold deposition and the influence of the additives are as sketched below,

Gold plating

i.   Choice of the electrolyte

Gold coatings prepared by electrolytic plating method have attracted much attention in the metal finishing industries.

Gold is used as a conducting material for interconnections and electrical contacts in the electronics industry. Gold cyanide is the most widely used gold complex in electro and electroless plating processes. The

disadvantages of using cyanide complexes are (i) the presence of cyanide in solution, wastewater, and the atmosphere raises environmental and safety concerns, (ii) the negative redox potential makes the formulation of electroless baths particularly difficult, (iii) the low exchange current density of cyanide complexes limit the rate of deposition, and (iv) the cyanide bath has problems of toxicity and suffers from the tendency of attacking the positive photoresist film that is used to delineate circuit patterns. So, the non-cyanide gold electroplating bath is chosen. In this context thiosulphate-sulfite acidic bath is to be tried with the aim of improving the performance the bath.

ii. Choice of the complexing and stabilizing agents

The non-cyanide baths containing either sulfite or thiosulphate as the complexing agent appear to be of limited in use because of insufficient stability of the systems. The sulfite-thiosulphate mixed ligands baths are more stable than sulfite or thiosulphate bath. It was found that the mixed ligand bath can be made further stable by the addition of some stabilizing agents.

In the present work, EDTA and citric acid are to be tried as the stabilizing agents. The addition of the stabilizer to the plating solution is to be controlled to such an extent that the bath can be operated at low pH values 3-6. The ability of the bath to function in the low pH range is significant because it enhances the compatibility with photoresists.

iii. Choice of the substrate

The gold plating is mostly done on copper substrates for microelectronic and decorative applications. In the present studies copper substrates with nickel strike coating are to be used.

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iv. Choice of the techniques

The chemical and electrochemical properties of the gold coatings developed are to be evaluated by different techniques. The cyclic voltammetry is to be performed in order to study the redox beahviour of gold deposition process. The electrochemical impedance is to be carried out to understand the charge transfer resistance accompanying the deposition process taking place in the interface.

The mechanical property like hardness in both asplated and heat-treated conditions are to be studied using Vickers hardness tester. The adhesion and porosity of the gold coatings are to be studied by using bend Test and electrograpy.

SEM, AFM and X-ray diffraction tools are to be adopted to examine the surface morphology and crystal structure of the electroplated gold. The elemental analysis of gold deposit is to be carried out through EDX techniques.

iv. Additives for gold plating solution

In many organic systems, the additives added to get desired properties, should the additive be a weak complexing, non-chelating addition agent that could take the place of the cyanide in the cyanide bath.

A through review of literature on addition agents in gold plating indicates numerous addition agents are employed to get desired structure with expected properties of gold coatings.

Phosphates, carbonates, acetates and citrates are commonly used as buffering and conducting agents for gold plating bath. In gold plating baths metals such aas Cd, Ti, Mo,W, Pb,Zn, Fe, In, Ni, Co, Sn, Cu, Mn and V in

various concentrations are used as brightening additives in cyanide bath. Few references are available for the use of some organic amines etc. In this investigate aniline, SLS, thiourea and pyridine are to be tried with a view to improve the bath properties as well as the deposit characteristics. Again as mentioned earlier all the techniques are to be tried to ascertain the properties.

Expected out come

The expected outcome of the studies is to identify a gold-thiosulphate-sulfite complex bath with judicious selection of stabilizing agents and proper additives. The bath is expected to give good throwing power, plating rate and current efficiency. While understanding the mechanism of the deposition process, structure and properties of the electrodeposits are to be monitored. Finally an inexpensive method of recovery of gold from plating waste is to be attempted.

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