Taguchi Design of Internal Stress and Friction Measurements during Electrodeposition

Ebru Saraloglu Güler* and Ishak Karakaya

1Department of Mechanical Engineering, Başkent University, Ankara, Turkey
2Department of Metallurgical and Materials Engineering, Middle East Technical University, Turkey

Abstract
The electroplating method is a promising alternative to produce composite plating by using dispersed fine particles in the metal plating bath. The process can be either called electro-deposition or composite deposition. The particles are trapped in the deposit during the process. Internal stress is a common problem in plated deposits that affect the performance of the coatings and may even result in adhesion problems. Hence, the amount and type (compressive or tensile) of the internal stress must be controlled. MoS2 particle – nickel coatings can be referred as self-lubricating coatings that satisfy the demand for decreased friction in severe applications. The internal stress during MoS2 particle – nickel electro-deposition was measured by deposit stress analyzer. The effects of electroplating parameters that are MoS2 particle concentration, temperature and coating thickness on the internal stress values were investigated by Taguchi design. It is found that increasing MoS2 particle concentration and coating thickness led to decrease in the internal stress developed during MoS2-nickel composite coatings. Interaction effects of these parameters were also revealed.

Keywords: Electro-deposition; Lubrication; Tensile

Introduction
Internal stress is one of the major problems encountered during deposition that can lead to detachment of the coating [1]. The deposit contracts or expands respect to the substrate due to the type of the stress that can be either tensile (contraction) or compressive (expansion) and denoted by + and - respectively [2]. The stress that is in the range of 30 MPa in electrodeposits up to 100 µm thickness can be considered as acceptable [3]. It was concluded that increasing the temperature decreased the internal stress [4,5]. The combination of parameters that produce minimum internal stress during Ni-MoS2 electro-co-deposition was investigated in this study. Since MoS2 has a self-lubricating property the deposition has the advantage of friction and high temperature applications. Therefore, it is critical to obtain stress free deposits or coatings with minimum stress level.

Taguchi design from Minitab was used to evaluate the influence of parameters and the interactions of these parameters during the experiments. Interaction parameter is important to observe the change of the response value by a specific parameter when the other parameter is high or low.

The easier and sufficiently precise measurement of internal stress can be done by the bent strip method. Two legged copper strips of 0.0508 mm thickness are immersed in the plating tank during bent strip testing method. Only one side of the strip is let to be plated by insulating the other side of the legs of the strips before deposition (Stein, 1996). Stress calculation is given as [2]:

\[
t = \frac{\Delta m}{D \times A \times 2.54}
\]

\[
S = \frac{U \times K}{3 \times t}
\]

Where: t: thickness of deposit (inch), Δm: weight gain (gram), D: density of nickel (g/cm^3), A: area (cm^2), S: stress (psi), U: number of increments, K: strip constant (psi x inch).

Experimental Section
The Watts bath containing 300 g/L NiSO4·6H2O (63035981; Unicore, Belgium), 50 g/L NiCl2·6H2O (7791-20-0; Selnic, France), and 40 g/L boric acid (minimum% 99.9 H3BO3, Etibank, Turkey) was prepared for the study. Following the bath preparation, the MoS2 particles with 1.440 µm are incorporated. Incorporation process involves 5 hours agitation of the particles in 50 ml Watts’s solution to make slurry and 1 hour agitation following the completion up to the desired volume [6]. Nickel anode (5 × 5 cm) was used. Copper alloy-194 bent strips received from Specialty Testing and Development Co. with a constant, K, of 0.3449 (psi x inch) were used as the cathode.

Figure 1: Copper strips having strip constant of 0.3449 with one side of the legs varnished.
(Figure 1) that were cleaned by alkaline immersion treatment before plating. Schematic drawing of the electroplating cell with nickel anode and copper strip is showed in Figure 2.

The electroplating parameters were set as MoS$_2$ particle concentration (10 and 30 g/l), temperature (30 and 50°C) and coating thickness (25 and 50 µm) with two levels. Internal stress will be the response value for Taguchi design given in Table 1. Two levels were designated by 1 and 2 for low and high values of each parameter respectively.

**Results and Discussion**

The internal stress values were measured for each experiment by bent strip method showed in Figure 3 and listed in Table 2 as the response value. The change in the internal stress with MoS$_2$ concentration, temperature and coating thickness is showed graphically in Figure 4. Increase in MoS$_2$ concentration and coating thickness decreased the internal stress whereas increasing temperature led to a slight increase in the internal stress. The result is consistent with the claim of Chou et al. [7] that is particle incorporation in nickel deposits has decreasing effect on internal stress. Moreover, thickness effect result also agrees with the study [8]. The interaction parameters were given in Figure 5. Increasing MoS$_2$ content transformed the temperature effect on the internal stress from increasing to decreasing (Figure 5a) and suppressed the coating thickness effect on the internal stress (Figure 5b). In addition, the regression equation for internal stress (S) is provided by the below equation showing the amount of the effects.

\[ S = 52.3 - 22.3A + 2.98B - 10.4C. \]

The most effective parameter is MoS$_2$ concentration according to the regression analysis since its coefficient is 22.3. The effect of temperature can be taken as insignificant that is supported by the result of the study of Schlesinger and Paunovic due to the factor of 2.98 [9]. On the other hand, some of the studies claim that increase in the temperature led to decrease in the internal stress [4,5,10].

![Figure 2: Schematic drawing of the cell showing nickel anode and Cu strip with one leg insulated cathode in watts bath.](image)

![Figure 3: Bent strip test analysis setup after experiment 3 given in Table 1 showing the number of increments (U).](image)

![Figure 4: Internal stress (MPa) versus A: MoS$_2$ concentration (g/l), B: Temperature (°C), C: Thickness (µm).](image)

![Figure 5: (a and b) Internal stress (MPa) versus interaction parameters of MoS$_2$ concentration, temperature and thickness.](image)

**Table 2: Internal stress values for the experiments listed in Table 1.**

| Experiment No | Response Internal Stress (MPa) |
|---------------|--------------------------------|
| 1             | 25.2                           |
| 2             | 2.3                            |
| 3             | 33.4                           |
| 4             | 14.6                           |
| 5             | -0.4                           |
| 6             | -2.1                           |
| 7             | -6.5                           |
| 8             | -4.6                           |

**Table 1: Taguchi design showing low (1) and high (2) values for each parameter during each experiment.**

| Experiment No | MoS$_2$ (g/l) | Temperature (°C) | Thickness (µm) |
|---------------|---------------|------------------|----------------|
| 1             | 1             | 1                | 1              |
| 2             | 1             | 1                | 2              |
| 3             | 1             | 2                | 1              |
| 4             | 1             | 2                | 2              |
| 5             | 2             | 1                | 1              |
| 6             | 2             | 1                | 2              |
| 7             | 2             | 2                | 1              |
| 8             | 2             | 2                | 2              |
Conclusion

MoS₂ concentration is the most important parameter that affects the internal stress. However, it must be at proper amount to show its effect on decreasing internal stress properly. Thick coatings led to decrease in the internal stress. Thickness effect on internal stress is more significant when the MoS₂ concentration is low and it van be negligible when MoS₂ concentration is increased to 30 g/l.

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