

Fig. B-1. Summary of round-robin analysis of excitation intensity dependence of PL intensity ratio of P-doped sample (81 Ω -cm). Excitation conditions I, II, and III denote excitation intensities of 100 mW, 50 mW, and 25 mW, respectively.

pass. Nonetheless, it remains rather difficult to fix the excitation intensity at a certain level.

To examine the variation of the PL intensity ratio induced by the difference in excitation intensity, we performed a round-robin analysis of the PL measurement for B-doped and P-doped standard samples under three excitation intensity conditions: the incident power was varied as 25, 50, and 100 mW. The PL intensity ratios of $P_{TO}(BE)/I_{TO}(FE)$ and $B_{TO}(BE)/I_{TO}(FE)$ for the P-doped sample, which is the same sample as in Fig. 4 and 5, under the three excitation conditions measured by six laboratories, are summarized in Fig. B-1.

As expected from the excitation intensity dependences of the free and bound exciton luminescence, the PL intensity ratio increases with the excitation intensity. However, it should be pointed out that the increment of the ratio, induced by an increase of the excitation intensity by a factor of 2, was less than 20% for both P and B impurities and for each laboratory. The relative standard deviation of the data taken under a certain excitation condition (25, 50, or 100 mW) by six laboratories was about 15%, while that of all the data taken under the three excitation conditions was about 20%. Similar results were obtained for the other standard samples. This result leads us to suggest that the deviation in the excitation intensity does not cause a serious error in obtaining the PL intensity ratio if the deviation is within a factor of 2.

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The Internal Stress in Ni, NiFe, CoFe, and CoNi Layers Measured by the Bent Strip Method

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ABSTRACT

The physical meaning and formulas for the calculation of the different, but interrelated, types of the internal stress (IS) in thin films (instantaneous, residual, and average) are reviewed. These formulas are then applied to the cases of plated Ni and NiFe, CoFe, and CoNi alloys. The IS profiles (the IS distribution through the film thickness) are given for all cases. The relationship between the IS and the structure parameter (phase composite, crystallite size, and microstain) changes with alloy composition is discussed.

During the deposition of thin or thick layers by electrochemical (1-7) or vacuum (8-10) methods, an internal stress is almost always developed. IS affects some of the most important physical, mechanical, and protective properties of electrodeposits, e.g., magnetic behavior, wear resistance, adhesion, fatigue strength, and corrosion resistance. High tensile IS can cause cracking of the coatings, thus modifying their protective properties. Cracks may be generated both during and after deposition, leading to a decrease in the fatigue strength of the entire system.

There is an increased interest in obtaining a more accurate characterization of the strained state of the total system, i.e., substrate and coating. It is required to determine not only the average stress in the coating, but also the residual stress distribution through its thickness; this is done by plotting the residual stress diagram (RSD) or stress profile (6). Thus, for example, if higher compressive stresses are present in the layers adjacent to the substrate, there is an increased probability of poor adhesion between the coating and the substrate. A knowledge of this stress distribution can also provide useful information on the mechanisms causing the stress.

Alloy electroplates from metals of the iron group are widely used as protective-decorative coatings. In addition, due to their ferromagnetic properties, these alloy coatings are used in the electronic industry. Since the chemical composition affects their phase composition, crystallite size, preferred orientation (texture), and IS, it thus also determines the magnetic properties, corrosion resistance, and electrical and mechanical behavior (11-13). Therefore, the deposition of layers with predetermined properties requires a reliable control both of the composition and structure, as well as IS.

The present paper outlines a method for the accurate and complete characterization of the strained state of the coating. The IS is measured for nickel and binary alloys of

metals from the iron group, using the bent strip method (1), and the relationships between the IS and structure are determined. The use of the dilatometric method for the IS measurement has been published previously (14-17).

Stress Types during the Deposition of the Coating

The mechanically strained state of the coating can be determined precisely by three typical stress patterns, namely, instantaneous, residual, and average. In order to provide a definition, we presume that the coating is deposited layer by layer (see Fig. 1). As a result of the deposition of the first layer with thickness Δt , an instantaneous stress, σ_{11} , is initiated in it, while in the substrate a reverse stress, σ_0 , is induced so as to balance the system. During the deposition of the second layer with an instantaneous stress, σ_{22} , the previously deposited layer is treated as a substrate, i.e., a reverse sign stress, σ_{12} , is induced in the first layer. Then the residual stress in the first layer after the deposition of the second layer can be presented as $\sigma_1 = \sigma_{11} + \sigma_{12}$. In a similar way the residual stress in the *i*-th layer after the deposition of the entire coating, comprising k layers, is as follows

$$\sigma_{i} = \sigma_{ii} + \sum_{j=i+1}^{k} \sigma_{ij}$$
 [1]

where σ_{ii} is the instantaneous stress, initiated in the *i*-th layer due to its deposition, while the second term takes into account the effect of other upper layers upon the strained state of the layer under consideration. The σ_i values represent the RSD during deposition.

The relationship between the residual stress in a given layer, (σ_i) , and that averaged through the thickness t, $(\overline{\sigma})$, is given by the mean value theorem

$$\overline{\sigma} = 1/t \int \sigma_i dt$$
 or $\overline{\sigma} \approx 1/t \sum_{i=1}^k \sigma_i \Delta t$ [2]

Equations [1]-[2] are general relationships and are not affected by the IS measuring method used. A planar strained state exists in electrodeposits, *i.e.*, two principal stresses act in each layer of the coating— σ_i^x and σ_i^y (18), their values being equal to each other (see Fig. 2). The presence of a planar strained state is taken into consideration by introducing into the formulas for IS the term $1/(1 - \nu)$ (19), where ν is Poisson's ratio.



Fig. 1. Instantaneous (σ_{ii}) and residual (σ_i) IS during deposition: 1. first layer; 2. second layer; k, the entire coating with k-layers deposited. For the sake of simplicity, we show a uniform distribution of instantaneous IS through the thickness.



Fig. 2. Schematic illustration of the stress distribution within the plane of the coating and through its thickness: 1. coating; 2. substrate.

Formulas for the Different Types of IS

In the presence of a tensile stress in the coating, there is a tendency to contraction and if we could imagine a separation of the coating from the substrate, it would have decreased its length (Fig. 3a-1). During the elastic interaction between the substrate and the coating, a pair of equal but opposite forces, F, which are trying to equalize the lengths of substrate and coating, are applied at different points, so that a bending moment M is initiated (Fig. 3a-2). The superposition of normal and bending stress patterns leads to the realization of a complex strained state of the system substrate-coating; compressive stress acts within the nearest to the coating layers of the substrate, while tensile stress is initiated in the more distant layers, *i.e.*, a zero stress layer (a neutral axis) exists in the substrate. The free end of the cathode is deviated from its initial position toward the anode. In the presence of compressive stress in the coating, the bending is in the opposite direction (Fig. 3b).



Fig. 3. Strained state of the substrate-coating system during the bent strip measurement: 1. the coating is imagined as being separated from the substrate; 2. elastic interaction between substrate and coating; (a) tensile IS; (b) compressive IS.

Different approaches for the determination of the instantaneous stress have been used (20-23), and various methods for the RSD plotting have been proposed (24-25). A comparison of these different methods has been published in Ref. (24); here we present only those relationships which have been used in the present paper.

For the instantaneous stress (σ_{ii}) evaluation (20-22).—

$$\sigma_{\rm ii} = \frac{\bar{E}_{\rm o} d^2}{3l^2} k_{\rm i} \frac{\Delta f_{\rm i}}{\Delta t}$$
^[3]

where Δf_i is the alteration of the cathode deviation due to the deposition of the *i*-th layer, Δt is the thickness of a thin layer ($\Delta t \rightarrow 0$), *d* is the thickness of the cathode, *l* its length, \overline{E}_o is the reduced Young's modulus of the substrate, *i.e.*, $\overline{E}_o = E_o/(1 - v_o)$

$$k_{i} = \frac{1 + 4\tilde{\gamma}\vartheta_{i} + 6\tilde{\gamma}\vartheta_{i}^{2} + 4\tilde{\gamma}\vartheta_{i}^{3} + \tilde{\gamma}^{2}\vartheta_{i}^{4}}{1 + 2\vartheta_{i} + \tilde{\gamma}\vartheta_{i}^{2}} \qquad [4]$$
$$\vartheta_{i} = \frac{t_{i}}{d} \qquad \tilde{\gamma} = \tilde{E}/\tilde{E}_{o}$$

where \tilde{E} is the reduced Young's modulus of the coating. Equation [3] is applied for real Δt values ($\approx 1 \ \mu m$) and this

approximation has been discussed in Ref. (24).

For the evaluation of the residual stress σ_i (24).—

$$\sigma_{1} = \frac{\overline{E}_{o}d^{2}}{3l^{2}} \left\{ k_{i} \frac{\Delta f_{i}}{\Delta t} - \sum_{j=i+1}^{k} \frac{k_{j}\Delta f_{j}}{d\overline{\gamma}^{-1} + t_{j-1}} - \frac{6\overline{\gamma}}{d^{2}} \left[\left(t_{i} - \frac{\Delta t}{2} \right) \sum_{j=i+1}^{k} + \Delta f_{j} + \sum_{j=i+1}^{k} C_{j}\Delta f_{j} \right] \right\} \quad [5]$$

$$c_{j} = \frac{d}{2} \left(\frac{1 - \overline{\gamma} \vartheta_{j}^{2}}{1 + \overline{\gamma} \vartheta_{j}} \right) \qquad [6]$$

where c_j is the position of the neutral axis as given by Popereka (20). Equation [6] is strictly correct only in the case of pure bending, since it does not take into consideration the shift of the neutral axis as a result of the action of the additional axial forces, but it still can be used [see Ref. (24)].

For the determination of the average stress $\bar{\sigma}$.—A critical review of the different equations for the evaluation of average stress can be found in Ref. (26) and an accurate version has been proposed, which we have used

$$\overline{\sigma} = \frac{\overline{E}_{o}d^{2}}{3l^{2}} \left(\frac{1+\overline{\gamma}\vartheta^{3}}{1+\vartheta}\right) \frac{f}{t} = \overline{\sigma}_{\text{Stoney}} \cdot \left(\frac{1+\overline{\gamma}\vartheta^{3}}{1+\vartheta}\right)$$
[7]

 $\overline{\sigma}_{\text{Stoney}}$ is a modified equation of Stoney (1) that takes into account the planar strained state (18-20). A more accurate relationship for the determination of $\overline{\sigma}$ is given in (25), but it requires more sophisticated calculations. In the presence of postplating alteration of IS for the determination of the average IS, $\overline{\sigma}^*$, use of Eq. [7] is recommended by replacing f with $f^* = f + f_{\text{postplating}}$.

Experimental Procedure

The cathode is fixed to the holder, parallel to the anode, (Fig. 4a) while the holder is housed in a double-thermostated electrolytic cell with circulation of the electrolyte (Fig. 4b). Electrodeposition is carried out only on the side toward the anode, while the other side is insulated with a suitable varnish. The following foils were used as substrates (elastic cathodes) for the various coatings: copper for CoNi, brass for CoFe, and platinum for nickel or NiFe. The use of platinum offers the possibility of increasing the reproducibility and accuracy of IS measurements by depositing onto the same substrate several times without replacing it in the holder and after the anodic dissolution in the same electrolyte. In this case, with 40 mm long and 0.2 mm thick cathode, the sensitivity is 0.05 kg/cm and takes into consideration the thickness of the coatings, their IS values, and the microscope magnification. Plating



Fig. 4. IS measurement by the bent strip method: (a) strip holder (viewed from the top); (b) the cell.

conditions are given in Table I. The alloy composition is given as a weight percentage.

Experimental Results and Discussion

Nickel.—During the deposition of nickel from electrolytes without organic additives (Table I), a uniform decrease of the instantaneous stress is observed as the coatings become thicker and the residual stress is rela-

Table I. Electrolyte composition and deposition c

	Electrolyte composition (g/liter)	T (°C)	Current density (A/dm ²)	pH
1.	Nickel NiSO ₄ \cdot 6H ₂ O— 262; NiCl ₂ \cdot 6H ₂ O— 50; H ₃ BO ₃ — 35; 2butyne— 1.4diol-0.2; saccharin— 1.5.	52	4.0	4.5
2.	Binary alloys 2.1. Nickel-iron (16.8-32.4% Fe) NiSO ₄ · $7H_2O$ — 80; NiCl ₂ · $6H_2O$ — 75; H ₃ BO ₃ — 40; FeSO ₄ · $7H_2O$ — 10-20; sodium citrate— 10; sodium gluconate— 10; saccharin— 4; NIFEBOM (27)— 2 ml/iter	65	5.0	3.0
	$\begin{array}{l} \text{A17 Encoded} \\ 2.2 \text{ Iron-cobalt} (32.75\% \text{ Fe}) \\ \text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 & -70.210; \\ \text{CoSO}_2 & \text{CO}_2 & \text{CO}_2 & \text{CO}_2 & \text{CO}_2 \\ \text{COSO}_2 & \text{CO}_2 & \text{CO}_2 & \text{CO}_2 & \text{CO}_2 \\ \text{COSO}_2 & \text{CO}_2 & \text{CO}_2 & \text{CO}_2 & \text{CO}_2 & \text{CO}_2 \\ \text{COSO}_2 & \text$	60	5.0	1.8
	2.3. Cobalt-nickel (0.40% Ni) $CoSO_4 \cdot 7H_2O = 280.84;$ $NiSO_4 \cdot 7H_2O = 0.196; H_3BO_3 = 30.$	80	3.0	5.0





Fig. 5. Instantaneous (σ_{ii}) and residual (σ_i) IS vs. thickness of nickel coatings deposited in Watts electrolyte. The $\overline{\sigma}$ value is calculated using Eq. [7].

tively homogeneously distributed (Fig. 5a). A similar type of behavior has been found by Wagner (6) who used an IS meter and a spiral contractometer.

The presence of 0.2 g/liter 2butyne-1,4diol (butynediol) in the electrolyte leads, as expected, to an increase of stress. However, no substantial alterations in the character of the stress-thickness relationships occur; the two curves merely shift to higher values (Fig. 5b). Similar results were obtained earlier using the dilatometric method for measuring IS; however, these results were for higher butynediol concentrations (0.3 g/liter) and different deposition conditions (15). The increase of stress in the presence of butynediol can be attributed to the combined effect of three factors, namely, (i) a reduced grain size (28, 29), (ii) enhanced hydrogenation of the coatings (30), and (*iii*) lateral growth of the crystallites (31).

The addition of 1.5 g/liter saccharin changes the shape of both curves in that when a coating thickness of 1 μ m is reached a sign reversal of the two stress types occurs. Thicknesses above 1 μ m display a uniform distribution of both σ_{ii} and σ_i (Fig. 5c). A transition from a large tensile stress to a compressive one in 0.8 μ m thick nickel coatings in the presence of saccharin only has been reported before (32) for the case where only the instantaneous stress was measured.

It is well documented that saccharin and other sulfurcontaining compounds reduce IS (2, 33-36). However, in all these studies, only the average stress and its relationship with the deposition conditions and surfactant concentrations have been investigated. During the deposition of nickel in the presence of saccharin, there is a decrease of the dimensions of coherent scattering domains (CSD) (37, 38), as well as an abrupt increase of the stacking fault concentration and dislocation density (38). It is considered that these structural changes are due to the adsorption and inclusion of sulfur in the crystal lattice of nickel (38) and along the grain boundaries (37, 38). Minimum dimensions of CSD and maximum values of the dislocation density and the mechanical strength of the coatings have been established in the simultaneous presence of saccharin and butynediol. It is generally accepted that saccharin decreases IS due to the restriction of lateral growth and inhibition of the coalescence of the separate islands, considering the initial deposition stages (39).

If we insert into Eq. [2] σ_i values calculated using Eq. [5], then we obtain $\overline{\sigma}$ values which are within 1-2% of those determined using Eq. [7]. A postplating alteration of IS was found only in coatings deposited in electrolytes containing both additives, *i.e.*, saccharin and butynediol. The absolute value of the compressive stress decreases after the current



Fig. 6. Average IS vs. iron content in the alloy



is switched off and remains constant at a value of -4.8 kg/mm^* , *i.e.*, the currentless change of the stress $\Delta \overline{\sigma}^* = 1.1 \text{ kg/mm}^2$. The reason for the appearance of postplating tensile stress is the evolution of hydrogen which has been codeposited in the coatings (16, 17, 40).

Nickel-iron.—NiFe alloys deposited in the electrolyte composition 2.1 (Table I) have the fcc structure (41). When the amount of iron increases from 0 to 28%, the size of the CSD decreases from 50 to 14 nm (42). Probably this reduction of crystallite size is the main reason for the increase found for $\bar{\sigma}$ with increase in iron content of the alloy (Fig. 6). A similar relationship has been found by other researchers (43, 44). As the iron content is further (>28%) increased, $\bar{\sigma}$ passes through a maximum in the region of 40-60% Fe (45).

The instantaneous IS values do not change significantly as the thickness of the alloy coating increases, whereas the residual stresses are always increased (Fig. 7a). The increase of iron content in the alloy does not change the character of these curves but only shifts them to higher stress values (Fig. 7b and c). Optimum data for strength and plasticity of the coatings have been obtained when the iron content is about 20% (42). By comparing these data with the IS results presented here, it can be concluded that alloy coatings, containing up to 20-25% iron display good mechanical properties.

Cobalt-iron.—The electrolyte composition is shown in Table I (2.2). The average IS decreases as the iron content in the alloy increases (Fig. 8). Within the composition range investigated (30-75% Fe) the coatings are single



Fig. 7. Instantaneous (σ_{ii}) and residual (σ_i) IS vs. coating thickness

phase with a BCC structure and an axial texture <111> (46).

The stress profiles show a decrease of both stress types $(\sigma_{ii} \text{ and } \sigma_i)$ up to coating thicknesses of 5-8 μ m (Fig. 9a). An increase in iron content in the alloy does not affect the shape of the curves but merely changes the values for both types of stress—the curves shift to lower values (Fig. 9b and c). On the basis of the relationships shown in Fig. 8 and Fig. 9, it can be concluded that critical stress levels, *i.e.*, values exceeding the mechanical strength value of the coating, are likely in very thin films (less than 1 μ m) and in alloys containing less than 35% iron.

Cobalt-nickel.—An investigation of the relationships between the structural parameters (phase composition, CSD



Fig. 8. Average IS vs. iron content in the alloy



size, and microstrain) and magnetic properties on one hand, and Ni content (0-40%), on the other, is reported in (47). The electrolyte was 2.3 as detailed in Table I. The results are summarized in Fig. 10.

The decrease of $\overline{\sigma}$ is probably due to the increase in CSD size; this is in agreement with data given by Doljack and Hoffman (48) and Kushner (49). The abrupt change in alloy phase composition as the nickel content exceeds 20% gives rise to an increase in $\overline{\sigma}$. The region of minimum stress values (11-18% Ni) is characterized by an abrupt decrease of the instantaneous and residual stress up to coating thicknesses about 10-20 µm (Fig. 11 a-b). Within the region of change in phase composition (24% Ni), the shape of the curves of both stress types is as follows: σ_{ii} is decreased, while σ_i , is relatively uniformly distributed (Fig. 11c). Further increases in Ni content lead to a change in the character of both relationships. The stress distribution through the coating thickness displays a complex character (Fig. 11d). A comparison of the curves in Fig. 11 shows that the CoNi alloy composition change affects not only the average stress, but the stress profile also.

For the composition range of 0-20% Ni, the amount of HCP phase, the microstrain and the coercivity all remain constant. The superposition of the IS change and the effect of other factors (phase composition, CSD size of both phases, and their texture correspondence) is the reason for the observed extreme value of H_c within a narrow concentration range (47) (Fig. 10).

Conclusions

1. The mechanically strained state of the coating during deposition is determined with the aid of three interrelated stress types: instantaneous (σ_{ii}), residual (σ_i), and average ($\overline{\sigma}$).

2. Butynediol increases the IS in nickel coatings but does not affect the instantaneous and residual stress distri-



Fig. 9. Instantaneous (σ_{ii}) and residual (σ_i) IS vs. coating thickness

bution through the coating thickness. The presence of saccharin in the electrolyte causes not only a stress sign reversal, but also alters the residual stress diagram (RSD).



Fig. 10. HCP phase content, coherent scattering domain size (D), microstrain ($\langle \epsilon^2 \rangle^{1/2}$), average IS ($\overline{\sigma}$), and coercivity (H_c) vs. nickel content in CoNi alloys.



Fig. 11. Instantaneous (σ_{ii}) and residual (σ_i) IS vs. CoNi coating thickness

3. For single-phase alloys NiFe (32-17% Fe)-fcc and CoFe (63-25% Co)-bcc, a gradual decrease of $\bar{\sigma}$ is observed as the Fe or Co content, respectively, is lowered. In both cases one is adding an element, Fe or Co, which in the pure elemental state does not have the crystal structure of the binary alloys. The change in $\bar{\sigma}$ in the CoNi alloys at a constant hcp/fcc phase ratio is more complex within the composition range 0-20% Ni.

4. The instantaneous and the residual stress distribution through the coating thickness retains its character when the chemical composition is changed in the single-phase alloys (NiFe and CoFe) or when the hcp/fcc ratio remains constant in CoNi alloy (Ni 0-20%). Within the composition range 20-32% Ni in CoNi, where there is an abrupt drop in the HCP phase content; there is also a change in the residual stress diagram (RSD).

A detailed consideration of the mechanisms producing IS is beyond the scope of this paper but is the topic of others (3, 5, 31). The role of the evolution of hydrogen that is included during the deposition of the coatings as the subsequent IS initiation is also considered in other publications (16, 17, 50). The thermal stress and the IS relaxation in amorphous NiP coatings are discussed in Ref. (51).

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