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Thermal and intrinsic stresses in single-crystal nickel films

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Abstract. The thermal and intrinsic stresses in single-crystal nickel films, in the thickness range 500–3000 Å, prepared by epitaxial growth onto the (100) cleaved surfaces of NaF, LiF, NaCl and MgO have been obtained. Calculated results from ferromagnetic resonance experiments show that the magnitudes and directions of the film stresses depend on the nature of the substrate conditions and on the sequential ordering of the deposited layers. The experimental results revealed that the thermal stresses, which are elastic and isotropic in character, are larger than the intrinsic stresses and can be of either compressive or tensile type. This type of stress disappears after the film is removed from the substrate. The present results also show that there is no single model which sufficiently accounts for the magnitudes of the intrinsic stresses in the strained films.

1. Introduction

It is well known that many thin-film materials, by whatever means they are produced, are in a state of internal stress. The existence of such stress is of considerable importance in the use of thin films in microcircuit technology. The stresses can cause substrate cracking, film rupture and in other cases cause the film itself to flake away from the substrate. They are also expected to affect the optical, electrical and magnetic film properties. For instance, the abnormal values in the magnetic parameters, such as the saturation magnetisation and the crystalline and the uniaxial anisotropy constants, are due to the internal film stresses. In those cases where the stresses can be released (e.g. by stripping from their substrates), these parameters usually revert to their normal values.

Due to the difficulties of measuring the stress in magnetic epitaxial films, most of the reported measurements of stress were conducted on polycrystalline films. Previous work [1–4] on single-crystal nickel films was also limited to films prepared under certain deposition conditions. The present study is conducted, therefore, on a large number of single-crystal films of nickel prepared under various deposition conditions to determine the magnitude and direction of the thermal and intrinsic stresses, by suitable ferromagnetic resonance experiments and analysis. The aim of the analysis is to examine whether the present results can be subsumed under the existing theoretical models.

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2. Experimental details

Using electron bombardment evaporation, single-crystal nickel films were obtained by epitaxial growth on freshly cleaved (100) faces of NaF, LiF, NaCl and MgO. For epitaxial growth it was necessary to hold the substrate temperature at 380 °C at least during film deposition. Evaporations were carried out at a pressure of (1–2) × 10^{-6} Torr and at a deposition rate of 6–12 Å s^{-1}. High-energy electron bombardment of the substrate was employed prior to and/or during evaporation—a procedure which has been shown to improve the epitaxial quality of the resultant film [5]. The thickness of the evaporated films, ranging from 500 to 3000 Å, was measured using a multiple-beam Fizeau interferometric system [6]. The films were circular in shape with a diameter of 5 mm.

Ferromagnetic resonance (FMR) measurememnts were carried out at about 17 GHz at room temperature using a microwave spectrometer with a standard TE_{011} rectangular reflection cavity. The sample was placed in the middle of one of the vertical walls of the cavity and the magnet rotated about a vertical axis ensuring that the static (DC) and radio frequency (RF) fields remained mutually perpendicular. Low-frequency field modulation (50 Hz) and phase-sensitive detection were employed which gave the first derivative of the power absorption. The strength of the magnetic field was measured by a Hall probe which provided servo-control of the magnet.

It should be emphasised that a large number of single-crystal nickel films have been prepared under a wide range of deposition conditions. The best of the reproducible results (20 samples) have been selected for the present investigation. It has been found that the reproducibility of the results depends critically on the deposition conditions of the prepared samples. For this purpose a special movable substrate holder with a shutter was designed so that 12 samples can be accommodated. In consequence, samples with identical deposition conditions (with one variable condition, e.g. the film thickness, the substrate material, or the period of the electron bombardment of the substrate) could be obtained for the FMR experiments and, consequently, for the study of their mechanical properties, and for structural analysis by electron microscopy and electron diffraction techniques.

3. Results and discussion

3.1. *Strain–stress formula*

Using well known expressions derived from a classical elasticity theory [7], which combine the principal strain ε_{ij} and the principal stress S_{ij} with the elastic constants E_{11} and E_{12} for a cubic crystal, the strain tensor ε_{ij} can be expressed as follows:

$$\varepsilon_x = E_{11}S_x + E_{12}S_y \tag{1}$$

$$\varepsilon_y = E_{12}S_x + E_{11}S_y \tag{2}$$

$$\varepsilon_z = E_{12}S_x + E_{12}S_y. \tag{3}$$

It is assumed in the above relations that the stress component normal to the film is zero, i.e. $S_z = 0$, because the film is constrained at the substrate but not constrained in the normal direction. This implies that the strain (ε_z) perpendicular to the plane of the film is a result of the Poisson contraction, set up normal to S_x and S_y. The thermal strain normal to the film in the cubic system has been calculated by Vook and Witt [8]. They

assumed, in addition to the assumption mentioned above, that the shear strains, normal and in the plane of the film, were zero.

We have assumed that the stresses are uniform, and thus, by symmetry, that $\varepsilon_x = \varepsilon_y$ and $S_x = S_y = S_t$. According to these boundary conditions, the strain–stress expression given by equations (1)–(3) will take the following form:

$$\varepsilon = S_t(E_{11} + E_{12}) \tag{4a}$$

where

$$E_{11} = (C_{11} + C_{12})/(C_{11} - C_{12})(C_{11} + 2C_{12})$$
$$E_{12} = -C_{12}/(C_{11} - C_{12})(C_{11} + 2C_{12}).$$

Using the available bulk values of C_{ij}, reported by Alers *et al* [9] for Ni measured at 27 °C ($C_{11} = 2.508 \times 10^{12}$ dyn cm^{-2}, $C_{12} = 1.500 \times 10^{12}$ dyn cm^{-2}), E_{11} and E_{12} have been calculated with the following results: $E_{11} = 7.219 \times 10^{-13}$ cm^2 dyn^{-1}; $E_{12} = -2.702 \times 10^{-13}$ cm^2 dyn^{-1}. Hence

$$\varepsilon = 4.517 \times 10^{-13} S_t. \tag{4b}$$

The strain resulting from the difference in the thermal expansion coefficients of the film and the substrate is given by [10]:

$$\varepsilon = (\alpha_f - \alpha_s)\Delta T \tag{5}$$

where α_f and α_s are the average coefficients of expansion for the film and the substrate, respectively, and ΔT ($= T_d - T_m$) is the temperature of the substrate during film deposition minus the temperature at measurement.

3.2. *Remarks on the thermal stress*

In the epitaxial growth it is necessary to hold the substrate temperature during deposition appreciably above room temperature. On cooling from the deposition temperature T_d to the measurement temperature T_m, thermal stress (which for the present samples is elastic and of the isotropic planar type) will develop because of the difference between the thermal expansion of the film and the substrate. This stress is given by [10]:

$$S_{th} = (\alpha_f - \alpha_s)Y_f(T_d - T_m) \tag{6}$$

where Y_f is Young's modulus for the film.

Equation (6) indicates that the thermal stress can be of either sign, and can be controlled by a judicious choice of substrate material and deposition temperature. Since the thermal expansion coefficients of NaF, LiF and NaCl are larger than that of the nickel [11], the thermal stress measured at room temperature is thus compressive (negative). However for a MgO substrate the stress is tensile (positive) since the thermal expansion coefficient of MgO is smaller than that of nickel (see table 1). It should be noted that equation (6) has been used to calculate the thermal stress in single-crystal nickel films and the results are depicted in table 1. Values of the coefficients of thermal expansion of nickel ($\alpha_{Ni} = 13.4 \times 10^{-6}$ °C^{-1}) and the substrates used were taken from reference [11] and the value of Young's modulus of nickel ($Y_{Ni} = 2.2 \times 10^{12}$ dyn cm^{-2}) was taken from reference [12].

Table 1. Thermal stresses in single-crystal nickel films.

Substrate material	Coefficient of thermal expansion (10^{-6} °C^{-1})	Thermal stress (10^9 dyn cm^{-2})	Type of stress
NaCl	40.0	22.41	Compressive
LiF	33.9	17.24	Compressive
NaF	33.0	16.51	Compressive
MgO	10.5	2.44	Tensile

Table 2. Stresses in strained single-crystal nickel films prepared under various deposition conditions.

Film No	Substrate material	Film thickness (Å)	Substrate temperature (°C)	Intrinsic stress (10^9 dyn cm^{-2})	Total stress (10^9 dyn cm^{-2})
1	NaF	850	380	10.71	−5.80
2	NaF[a]	850	380	9.54	−6.97
3	NaF[a]	1540	400	11.34	−5.17
4	NaF[a]	1540	400	10.22	−6.29
5	LiF	2500	400	13.38	−3.89
6	LiF[a]	2500	400	11.34	−5.93
7	NaCl[a]	850	380	16.83	−5.58
8	NaCl[a]	850	380	15.23	−7.18
9	MgO	2500	400	−3.27	0.83
10	MgO[a]	2500	400	−2.93	0.49

[a] Electron bombardment was employed. Substrates 4 and 8 were bombarded for longer times than substrates 3 and 4.

3.3. *Intrinsic stress*

The contribution of this type of stress to the total film stress occurs mainly during film growth. It is rather difficult to interpret the nature of the stresses generated during the film formation processes, except in the most general terms. However, in order to obtain more information about the intrinsic film stress, whether compressive or tensile, the total film stress has to be calculated. Following Macdonald's resonance equation [13] for a strained single-crystal sample, Ahmad [14] has deduced formulae for calculating the total film stress as follows:

$$S_t = 4\pi M_0(M_e - M_0)/(2h_1 + 4h_3 + 8h_4/3) \qquad (7)$$

where M_0 is the saturation magnetisation of the strain-free film, M_e is the effective saturation magnetisation of the strained film and h_1, h_3 and h_4 are the magnetoelastic coefficients of the film. The values of the coefficients h_1, h_3 and h_4 have been taken from Ahmad *et al* [3]. Equation (7) has been used to calculate the total stress and the results are listed in tables 2 and 3.

It should be noted that the sign of the bracket ($M_e - M_0$) in equation (7) depends on the magnitudes of M_e and M_0 which have been obtained from the FMR measurements.

Table 3. Stresses in strained and strain-free single-crystal nickel films prepared under various deposition conditions.

Film No	Substrate material	Substrate temperature (°C)	Film thickness (Å)	Intrinsic stress (10^9 dyn cm^{-2})	'Total stress' (10^9 dyn cm^{-2}) Strained	Strain-free
11	NaF	400	1000	9.57	−6.94	0.39
12	NaF	380	3000	12.77	−3.74	0.13
13	NaCl	380	2600	19.68	−2.73	0.43

The FMR analysis has indicated that $M_e > M_0$ for Ni films deposited onto NaF, LiF and NaCl substrates, whereas $M_e < M_0$ for Ni films deposited onto MgO substrates. More specifically, the value of the effective magnetisation M_e of the present films was found to fluctuate between 404 to 794 G. We ascribed these fluctuations to the differences in the direction and magnitude of the isotropic stress in the films. Nevertheless, the average value of the saturation magnetisation M_0 calculated for different strain-free films was found to be equal to 492 ± 5 G, which is in good agreement with that from previous investigations [3, 4, 15]. In addition, the average value of the spectroscopic splitting factor (g) was found to be 2.15 ± 0.02 for the strain-free films, which is also in good agreement with previous measurements [3, 4]. A method for the calculation of M_e or M_0 and the g-factor from FMR measurements was described elsewhere [16].

The magnitude of the intrinsic stress can be obtained by subtracting the magnitudes of the thermal stress S_{th} from the total stress S_t. The resulting equation expressed as

$$S_{in} = S_t - S_{th}. \tag{8}$$

has been used to calculate the intrinsic film stress in strained films, and the results are illustrated in tables 2 and 3. The most important results which are illustrated in these tables can be summarised as follows. (i) The magnitude of the total film stress for electron-bombarded films deposited onto NaF, LiF and NaCl substrates is relatively larger than that for non-bombarded ones, and it is also larger for those bombarded for a longer rather than a shorter time. Conversely, films deposited onto MgO substrates show less stress for electron-bombarded films as opposed to the non-bombarded ones. But is should be noted that the stress in the former case is compressive, whereas in the latter it is tensile. (ii) The intrinsic stress for Ni films deposited onto all of the substrates used is relatively smaller for the electron-bombarded than for non-bombarded or less bombarded films. (iii) The residual stress of the strain-free films is about 10 times smaller than that of the strained films. Furthermore, the nature of the stress is changed from compressive for strained films to tensile for strain-free ones. (iv) Although the intrinsic stresses for most of the films are quite high, some of them have a value exceeding the elastic limit of the nickel bulk [17] (13.1×10^9 dyn cm^{-2}); such large stresses are in accord with other data on thin films [3, 18]. However, in compensation, the effect of the thermal stress will prevent the total film stress from reaching the yield point.

Another contribution to the intrinsic stress comes from the lattice misfit (η) between the nickel films and their substrates. Since the lattice constant of the nickel is less than that of all of the substrates used, one would expect that a large portion of this stress

Table 4. Characteristics of nickel films deposited onto various crystalline substrates.

Deposit	Substrate material	Structure type	Cleavage plane	Lattice constant[a] (Å)	Misfit (%)
Ni	NaCl	FCC	(100)	5.64	38
Ni	NaF	FCC	(100)	4.63	24
Ni	MgO	FCC	(100)	4.21	16
Mi	LiF	FCC	(100)	4.02	12

[a] Reference [19].

Table 5. Stresses in low-strain single-crystal nickel films. The deposition conditions are as follows: substrate temperature, 400 °C; film thickness, 2500 Å for films number 14–17 and 2900 Å for films number 18–20; thickness of NaF, 400 Å.

Film No	Substrate material	Films	Intrinsic stress (10^9 dyn cm^{-2})	Total stress (10^9 dyn cm^{-2})	
				Strained	Low-strain
14	NaCl	NaF:Ni	13.39	−3.12	−0.27
15	NaCl	Ni:NaF	16.02	−0.49	0.77
16	NaCl	NaF:Ni:NaF	17.10	0.59	0.33
17	LiF	NaF:Ni	17.40	0.13	
18	LiF	Ni:NaF	12.59	−4.68	
19	MgO	NaF:Ni	−0.35	2.09	
20	MgO	Ni:NaF	−0.78	1.66	

would be taken up in the growth structure of the film through the lattice misfit. The lattice misfit between the film a_f and the substrate a_s is given by

$$\eta = (a_s - a_f)/a_s. \tag{9}$$

For the substrate used in the present work, and using equation (9), a large misfit is expected to occur when nickel is deposited onto NaCl substrates, with a lower misfit on LiF substrates (see table 4). A detailed analysis of the intrinsic stress due to the lattice misfit will be given in § 3.5.

3.4. Effect of NaF layers

In this section we shall consider the effect of the deposition of a NaF film prior to and/or after film deposition and the effect of the original substrate on the residual film stress. The total and intrinsic stresses of the films were calculated with the help of the equation used for the films discussed in § 3.3. The results of the calculations are presented in table 5.

Despite the stress in the low-strain film number 14 (Ni : NaF) being 10 times smaller than that of the same film in the strained case (Ni : NaF : NaCl), its sign, which indicates compression, does not change. Low strain is a convention used to refer to those samples which have been removed from their original substrates. On changing the deposition

sequence of the NaF film, it is expected that a different type of stress will result. Indeed, the state of the stress of the low-strain film number 15 (NaF : Ni) changes from compressive to tensile. It should be noted that when the film 15 (NaF : Ni : NaCl) is removed from the original substrate NaCl, the free surface of the nickel film is exposed to the atmosphere and so it may be oxidised. Thus, the NaF film for the above samples can be regarded as a protective layer against the inevitable oxidation of the free surface of the nickel films.

The stress in the strained film number 16 (NaF : Ni : NaF : NaCl) and of the same film in the low-strain case (NaF : Ni : NaF) shows different behaviour to the films described above: while the stress in both systems is a tensile type, the magnitude of the stress in the former system is larger than that in the latter. This result is consistent with those for the films illustrated in table 3. The stress in film numbers 17 (Ni : NaF : LiF) and 18 (NaF : Ni : LiF) is also different in nature and magnitude from that discussed above and this is due to the effect of the NaF film (see table 5).

The stress in film numbers 19 (Ni : NaF : MgO) and 20 (NaF : Ni : MgO) also shows a behaviour different from those of all the previous films. That is, the magnitude of the total stress in these films, shown in table 5, is much larger than that of the same type of stress in single films 9 and 10 (i.e., Ni : MgO) of table 2. Conversely, the magnitude of the intrinsic stress in films 19 and 20 is much smaller than that for the same type of stress in films 9 and 10. This is also due to the effect of the NaF film. It should be noted that the LiF and MgO crystalline substrates are almost insoluble in water and, so, it is difficult to remove the film from them. This is the reason why the sixth column of table 5 is incomplete. Also to be noted is the fact that the majority of the films presented in table 2 display total stresses which are relatively higher than those of the films presented in table 5, in addition to their nature being different. In other words, the decrease in the total film stresses (from 10^9 to 10^8 dyn cm^{-2}) and the changes in their sign from compressive to tensile, or vice versa, take place when the nickel film is coated by a NaF film.

3.5. The present results as compared with various stress models

A large number of models have been proposed to explain the intrinsic stress in thin films. It is appropriate to indicate briefly which of these models may be important in connection with the present data. The Murbach and Wilman model [20] cannot explain the present results, because it assumes that the substrate temperature reaches the recrystallisation temperature, which is about 530–600 °C for a deposition temperature of 380–500 °C. Since the epitaxial temperature for our films is about 400 °C, this would mean that the film temperature will rise by about 130 °C during deposition. Due to the thermal radiation of the evaporation source and latent heat of the deposition vapour beam, the effective substrate temperature at the surface can be higher than the average temperature measured by a thermocouple. Observations by other researchers [21–23] have confirmed this but they have shown that there is very little rise of the film temperature at the substrate, and any heat obtained from condensation of individual atoms will be dissipated very rapidly. In addition, it must also be noted that the Murbach and Wilman model predicts a constant tensile stress in continuous films which is independent of film thickness. In the light of the previous [18] and present results, compressive stresses have also been observed in different single-crystal films prepared under different deposition conditions. It is, therefore, concluded that such a model is inapplicable to the present results.

The amorphous–crystalline phase-change model [24] cannot be applied to the present results either, because such a model deals with a phase transformation from one form to another whereas the present films are of a single crystal of only single phase, namely face-centred cubic. In order to test the validity of the vacancy migration model [25], it would be necessary to prepare a number of films representing different stages of formation of the film and then to make FMR measurements on them to calculate their stresses. On the other hand, these films need to be annealed in order that the stresses can be calculated, to establish whether there is any difference between the measurements before and after the annealing process. In fact, neither were the films at the early stages of formation studied, nor was the annealing procedure accomplished. We are, therefore, forced to exclude this model from the present analysis.

The crystalline boundary relaxation model [26] also explains film stresses at early stages of film formation. In addition, this model depends on certain parameters such as crystallite dimension and average atomic relaxation distances which are not available for the present samples. But it should be noted that the presence of crystal imperfections resulting from the deposition process are more likely to be one of the permanent contributions to the intrinsic stress. This model, however, predicts little stress for single-crystal films. It is deemed inappropriate for explaining the present intrinsic stress. The electrostatic force model [27], which has been applied to discontinuous films, predicts very small stresses, and so is likewise inapplicable to continuous films and, hence, to the present films. Nevertheless, we have demonstrated in table 2 that all the films which have been exposed to electron bombardment show a larger stress than that of the non-bombarded or less-bombarded films. This means that the bombarded films are more strained with respect to their substrate than the non-bombarded ones. Since the stress of the non-bombarded films is of the same order of magnitude as those of the bombarded ones, the above model cannot account for the present values of the film stresses.

The surface tension model [28] has been found [29, 30] adequate for evaluating the intrinsic stresses in continuous and discontinuous films because it predicts the right magnitudes and directions of these stresses. Heavens and Smith [29] have shown that if the continuous film consists of numerous crystallites of uniform shape which are separated by a small distance, cohesive forces will be developed. Such forces will give rise to a tensile stress and it will decrease with increasing void content in the film. The influence of gas adsorption on the surface tension of the polycrystalline nickel films at room temperature has been investigated by Janssen [30]. The surface tension model seems to provide an explanation for the magnitudes and directions of the internal stresses in his samples. However, our preliminary analysis shows that the surface tension would have to be of the order 10^4–10^5 dyn cm^{-1} to explain the experimental results, whereas the value quoted [31] for clean polycrystalline nickel at room temperature is 2500 dyn cm^{-1}. In other words, the calculated intrinsic stresses for the present strained films based on the surface tension model [28] are approximately of the order of 10^8 dyn cm^{-2}. This value is much smaller than the magnitude of all the stresses of the strained films which are listed in tables 2 and 3. Nevertheless, it is much closer to our experimental results than the value which is predicted by all the other models previously mentioned.

Van der Merwe proposed a model [32] for the thin-film stresses caused by the deposition of a single-crystal film onto a single-crystal substrate. He has shown that strain can result from the misfit of films growing on single-crystal substrates which will be partially relieved by the formation of interfacial dislocations. He categorised the deposited films into two types; the first is for films which are compressed so that the

lattice constant matches that of the substrate. This type of film is not applicable to our films because it assumes no misfit at the film–substrate interface which is contrary to our present results, and so does not predict any interfacial dislocations. The second type deals with the case of a large misfit, which means that interfacial dislocations do exist at the film–substrate interface. These dislocations would decrease the lattice misfit at the expense of introducing an elastic strain into the film. Furthermore, according to this model compression or tension should be observed depending on the condition of negative or positive misfit; this agrees with the present results.

Preliminary analysis of the strained films showed that the experimental value of the strain falls into two categories: (i) for the low-strain and strain-free films it is 10 times greater than the value predicted by the interfacial model [32]; (ii) for the strained films it is 100 times greater than the value predicted by the same model. In other words, the percentage of the misfit accommodated by strain is much larger than the predicted value [32]. The elastic strain ε_e was obtained from the expression $\varepsilon_e = (a_f - a_0)/a_0$, where a_f and a_0 are the lattice constants of the strained and bulk value of the film, respectively. It should be noted that the lattice misfit of the Ni : NaCl system is relatively larger than the misfit of the Ni : NaF system. It is therefore expected that the intrinsic stress will be large in the former system and smaller in the latter. Accordingly the stress in the Ni : LiF system is expected to be between the above cases. Indeed, the calculated intrinsic stress for the Ni : LiF system, which is presented in table 2, is consistent with the argument given above and thus validates the interfacial misfit model [32].

4. Conclusions

Calculation of the stresses from FMR data in nickel films grown epitaxially on various crystalline substrates under closely controlled conditions, yielded the following experimental results.

(i) It is possible to obtain a quantitative separation between the intrinsic and thermal components of the stresses. The isotropic planar thermal stress is often large and is more predominant than the intrinsic one. It can frequently be controlled by a sensible choice of substrate material and deposition temperature. This type of stress can be eliminated by floating off the film from its substrate.

(ii) Deposition of a NaF film prior to or after nickel leads to the film being relatively much less strained than the one deposited directly onto the substrate. Consequently, the sign and magnitudes of the film stresses turn out to be different in these two cases. In addition, the NaF film was found to function to some extent as a protection from oxidation at the free surface.

(iii) It is rather difficult to specify a model that could represent quantitatively the magnitude of the intrinsic film stress. It seems, however, that this type of stress would appear to be best explained by a combination of the interfacial dislocation and the surface tension models. On the other hand, a reliable quantitative prediction for this particular stress has yet to be carried out.

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