R. S. Smith

# Measurement of Crystallite Size and Strain of Electroplated Films

Determination of crystallite size and strain in permalloy films is being undertaken as part of a research program to relate the magnetic properties of electroplated films to their structural properties.

The literature[1] reports the extensive use of x-ray diffraction techniques to determine the average crystallite size and rms strain on bulk quantities of polycrystalline materials. These techniques require measurements on several orders of a given family of lines. However, this technique cannot be applied to electroplated permalloy films, since only one line is available from a given family of lines for many of these thin samples.

The purpose of this note is to describe an analysis based on an approximation to the more sophisticated technique of determining multiple profiles. The method described here has yielded significant parameters for describing Fe-Ni samples.

## Procedure

An x-ray spectrometer is used to obtain a diffraction profile. With this apparatus we obtain a plot of the intensity, $I$, vs Bragg angle, $2\theta$, about some desired peak. The shape of this curve is determined by a number of factors, which include machine broadening, broadening due to small crystal size, and broadening due to rms strain.

In order to determine crystallite size and rms strain from the single line, the shape of the line must first be corrected for machine broadening of the beam. This is accomplished using the well known method of Stokes[2], by which a Fourier transform of the sample is modified by comparison with the transform of a standard specimen consisting of large, well annealed samples. The computations are performed in this laboratory on the IBM 704. The corrected Fourier coefficients $A_n$ are defined by:

$$I'(\theta) = \sum_{n=0} A_n \exp(2\pi i n x/a) ,$$

where the values of $A_n$ have been normalized so that $A_0=1$. Here,

$\theta \equiv$ angle of incidence of x-ray beam,
$\theta_0 \equiv$ angle of incidence of center of gravity,
$\theta_0-\theta_m \equiv$ the period of expansion of the Fourier series,
$\lambda \equiv$ wavelength of $x$ radiation,
$a = \lambda/[4(\sin\theta_m - \sin\theta_0)]$,
$x = (\sin\theta_0 - \sin\theta)(2a/\lambda)$.

Bertaut[3] has related $A_n$ to $D$, crystallite size; $l$, order; and $Z_n$, the relative displacement, in units of the cell parameter of two cells $n$ cells apart, in a direction parallel to the diffraction vector. When $n$ is small, $Z_n \cong n\varepsilon$, where $\varepsilon$ is the strain. If, in addition, it is assumed that throughout the crystal the dispersion about $\varepsilon$ over small distances is small, then one can state Bertaut's equation as:

$$A_n = \left(1-\frac{an}{D}\right)\cos(2\pi l\langle\varepsilon\rangle n) . \qquad (1)$$

Here $D$ represents the crystallite size, measured perpendicular to the reflecting lattice planes. The value $\varepsilon$ is the rms strain normal to the planes giving the x-ray peak, and $l=2a\sin\theta_0/\lambda$. Justification for this approximation to Bertaut's formula is based on the agreement between the experimental curves and the theoretical points presented in Fig. 1.

To determine $\varepsilon$ and $D$ from (1), conventional procedure requires measurements on more than one member of a family of lines. It will be shown, however, that equivalent values of $\varepsilon$ and $D$ can be obtained from measurements on single lines.

After the computed values of $A_n$ vs $n$ are obtained from the IBM 704, the procedure is to look up values of $D/a$ and $\varepsilon$ which best satisfy the equation

$$A(n) = \left(1-\frac{an}{D}\right)\cos(2\pi l\varepsilon n) . \qquad (2)$$

This operation has been facilitated by the use of Table 1, which is a compilation of $(1-x)\cos\theta$ for a series of values of $X$ and $\theta$.

Values of $X$ and $\theta$ are determined from the table which satisfy:

$$A_{1M} = (1-XM)\cos\theta M$$

$$A_{2M} = (1-2XM)\cos 2\theta M .$$

Of course, $A_M$ and $A_{2M}$ are two amplitudes that are chosen arbitrarily from the curve of computed values of $A_n$ vs $n$; then the strain, $\varepsilon$, and crystallite size are computed from

$$MX = a/D \qquad \text{and}$$

$$M\theta = 360 l\varepsilon . \qquad (3)$$

*Table 1* **Compilation of (1 − x) cos θ for a series of values of X and θ.**

| θ | (1−x) → | 0.99 | 0.98 | 0.97 | 0.96 | 0.95 | 0.94 | 0.93 | 0.92 | 0.91 | 0.90 | 0.89 | 0.88 | 0.87 | 0.86 | 0.85 | 0.84 | 0.83 | 0.82 | 0.81 | 0.80 | 0.79 | 0.78 | 0.77 | 0.76 | 0.75 | 0.74 | 0.73 | 0.72 | 0.71 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 99985 | 9898 | 9798 | 9698 | 9598 | 9498 | 9398 | 9298 | 9198 | 9098 | 8998 | 8898 | 8798 | 8698 | 8598 | 8498 | 8398 | 8298 | 8198 | 8098 | 7998 | 7898 | 7798 | 7698 | 7598 | 7498 | 7398 | 7298 | 7198 | 7098 |
| 2 | 99939 | 9894 | 9794 | 9694 | 9594 | 9494 | 9394 | 9294 | 9194 | 9094 | 8994 | 8895 | 8795 | 8695 | 8595 | 8495 | 8395 | 8295 | 8195 | 8095 | 7985 | 7885 | 7775 | 7675 | 7595 | 7495 | 7395 | 7295 | 7195 | 7095 |
| 3 | 99863 | 9886 | 9786 | 9686 | 9586 | 9486 | 9387 | 9287 | 9187 | 9087 | 8987 | 8888 | 8788 | 8688 | 8588 | 8488 | 8388 | 8289 | 8189 | 8095 | 7995 | 7895 | 7795 | 7695 | 7590 | 7489 | 7389 | 7289 | 7190 | 7090 |
| 4 | 99756 | 9876 | 9776 | 9676 | 9576 | 9476 | 9377 | 9277 | 9177 | 9077 | 8978 | 8878 | 8778 | 8678 | 8579 | 8479 | 8379 | 8279 | 8179 | 8080 | 7980 | 7880 | 7780 | 7681 | 7581 | 7481 | 7381 | 7282 | 7182 | 7082 |
| 5 | 99619 | 9862 | 9762 | 9663 | 9563 | 9463 | 9364 | 9264 | 9164 | 9065 | 8965 | 8866 | 8766 | 8666 | 8567 | 8467 | 8367 | 8268 | 8168 | 8069 | 7969 | 7869 | 7770 | 7670 | 7571 | 7471 | 7371 | 7272 | 7172 | 7072 |
| 6 | 99452 | 9845 | 9745 | 9646 | 9547 | 9447 | 9348 | 9249 | 9149 | 9050 | 8950 | 8851 | 8751 | 8652 | 8552 | 8453 | 8353 | 8254 | 8155 | 8055 | 7956 | 7856 | 7757 | 7657 | 7558 | 7458 | 7359 | 7259 | 7160 | 7060 |
| 7 | 99255 | 9826 | 9726 | 9627 | 9528 | 9429 | 9329 | 9230 | 9131 | 9032 | 8932 | 8833 | 8734 | 8635 | 8535 | 8436 | 8337 | 8238 | 8138 | 8039 | 7940 | 7841 | 7741 | 7642 | 7543 | 7444 | 7344 | 7245 | 7149 | 7047 |
| 8 | 99027 | 9803 | 9704 | 9605 | 9506 | 9407 | 9308 | 9209 | 9110 | 9011 | 8912 | 8813 | 8714 | 8615 | 8516 | 8417 | 8318 | 8219 | 8120 | 8021 | 7922 | 7823 | 7724 | 7625 | 7526 | 7427 | 7327 | 7228 | 7129 | 7030 |
| 9 | 98769 | 9778 | 9679 | 9580 | 9481 | 9383 | 9284 | 9185 | 9086 | 8987 | 8889 | 8790 | 8691 | 8592 | 8494 | 8395 | 8296 | 8197 | 8099 | 8000 | 7901 | 7802 | 7703 | 7605 | 7506 | 7407 | 7308 | 7210 | 7111 | 7012 |
| 10 | 98481 | 9749 | 9651 | 9552 | 9454 | 9355 | 9257 | 9158 | 9060 | 8961 | 8863 | 8764 | 8666 | 8567 | 8469 | 8370 | 8272 | 8173 | 8075 | 7976 | 7878 | 7779 | 7681 | 7583 | 7484 | 7386 | 7287 | 7189 | 7090 | 6992 |
| 11 | 98163 | 9717 | 9619 | 9521 | 9423 | 9325 | 9227 | 9129 | 9030 | 8932 | 8834 | 8731 | 8633 | 8534 | 8436 | 8338 | 8240 | 8142 | 8044 | 7946 | 7848 | 7750 | 7652 | 7553 | 7455 | 7362 | 7264 | 7165 | 7067 | 6969 |
| 12 | 97815 | 9684 | 9586 | 9488 | 9390 | 9292 | 9194 | 9096 | 8998 | 8901 | 8803 | 8705 | 8607 | 8509 | 8412 | 8314 | 8216 | 8118 | 8020 | 7923 | 7825 | 7727 | 7629 | 7531 | 7433 | 7336 | 7238 | 7140 | 7042 | 6944 |
| 13 | 97437 | 9645 | 9548 | 9450 | 9353 | 9256 | 9159 | 9061 | 8964 | 8866 | 8769 | 8671 | 8574 | 8477 | 8379 | 8282 | 8184 | 8087 | 7989 | 7892 | 7794 | 7697 | 7600 | 7502 | 7405 | 7307 | 7210 | 7112 | 7015 | 6918 |
| 14 | 97030 | 9605 | 9508 | 9411 | 9314 | 9217 | 9120 | 9023 | 8926 | 8829 | 8732 | 8635 | 8538 | 8441 | 8344 | 8247 | 8150 | 8053 | 7956 | 7858 | 7762 | 7665 | 7568 | 7471 | 7374 | 7277 | 7180 | 7083 | 6986 | 6889 |
| 15 | 96593 | 9562 | 9466 | 9369 | 9272 | 9176 | 9079 | 8983 | 8886 | 8789 | 8693 | 8583 | 8493 | 8402 | 8306 | 8209 | 8113 | 8016 | 7920 | 7823 | 7727 | 7630 | 7534 | 7437 | 7341 | 7244 | 7147 | 7051 | 6954 | 6858 |
| 16 | 96126 | 9516 | 9420 | 9334 | 9228 | 9131 | 9035 | 8939 | 8843 | 8747 | 8651 | 8555 | 8459 | 8362 | 8266 | 8170 | 8074 | 7978 | 7882 | 7786 | 7690 | 7593 | 7497 | 7401 | 7305 | 7209 | 7113 | 7017 | 6921 | 6824 |
| 17 | 95630 | 9467 | 9371 | 9276 | 9180 | 9084 | 8989 | 8893 | 8797 | 8702 | 8606 | 8511 | 8415 | 8319 | 8224 | 8128 | 8032 | 7937 | 7841 | 7746 | 7650 | 7554 | 7459 | 7363 | 7267 | 7172 | 7076 | 6980 | 6885 | 6789 |
| 18 | 95106 | 9415 | 9320 | 9225 | 9130 | 9035 | 8939 | 8844 | 8749 | 8654 | 8559 | 8464 | 8369 | 8274 | 8179 | 8084 | 7988 | 7893 | 7798 | 7703 | 7608 | 7513 | 7418 | 7323 | 7228 | 7132 | 7037 | 6942 | 6847 | 6752 |
| 19 | 94552 | 9360 | 9266 | 9171 | 9077 | 8982 | 8887 | 8793 | 8698 | 8604 | 8509 | 8415 | 8320 | 8226 | 8131 | 8036 | 7942 | 7847 | 7753 | 7658 | 7564 | 7469 | 7375 | 7280 | 7185 | 7091 | 6996 | 6902 | 6807 | 6713 |
| 20 | 93969 | 9302 | 9208 | 9115 | 9021 | 8927 | 8833 | 8785 | 8676 | 8566 | 8457 | 8363 | 8269 | 8175 | 8081 | 7987 | 7893 | 7799 | 7705 | 7611 | 7517 | 7423 | 7329 | 7235 | 7141 | 7047 | 6953 | 6859 | 6765 | 6671 |
| 21 | 93358 | 9242 | 9149 | 9055 | 8962 | 8869 | 8775 | 8682 | 8588 | 8495 | 8402 | 8308 | 8215 | 8122 | 8028 | 7935 | 7842 | 7748 | 7655 | 7561 | 7468 | 7375 | 7281 | 7188 | 7095 | 7001 | 6908 | 6815 | 6721 | 6628 |
| 22 | 92718 | 9179 | 9086 | 8993 | 8900 | 8808 | 8715 | 8622 | 8530 | 8437 | 8344 | 8251 | 8159 | 8066 | 7973 | 7881 | 7788 | 7695 | 7602 | 7510 | 7417 | 7324 | 7232 | 7139 | 7046 | 6953 | 6861 | 6768 | 6675 | 6582 |
| 23 | 92050 | 9113 | 9020 | 8929 | 8837 | 8744 | 8652 | 8560 | 8468 | 8376 | 8284 | 8192 | 8100 | 8008 | 7916 | 7824 | 7732 | 7640 | 7548 | 7456 | 7364 | 7271 | 7179 | 7087 | 6995 | 6903 | 6811 | 6719 | 6627 | 6535 |
| 24 | 91355 | 9044 | 8952 | 8861 | 8770 | 8678 | 8587 | 8496 | 8404 | 8313 | 8221 | 8130 | 8039 | 7947 | 7856 | 7765 | 7673 | 7582 | 7491 | 7399 | 7308 | 7217 | 7125 | 7034 | 6942 | 6851 | 6760 | 6668 | 6577 | 6486 |
| 25 | 90631 | 8972 | 8881 | 8791 | 8700 | 8610 | 8519 | 8428 | 8338 | 8247 | 8156 | 8066 | 7975 | 7884 | 7794 | 7703 | 7613 | 7522 | 7431 | 7341 | 7250 | 7159 | 7069 | 6978 | 6887 | 6797 | 6706 | 6616 | 6525 | 6436 |
| 26 | 89879 | 8892 | 8808 | 8718 | 8628 | 8538 | 8448 | 8358 | 8269 | 8178 | 8089 | 7999 | 7909 | 7819 | 7729 | 7639 | 7549 | 7459 | 7370 | 7280 | 7190 | 7100 | 7010 | 6920 | 6830 | 6740 | 6651 | 6561 | 6471 | 6381 |
| 27 | 89101 | 8820 | 8732 | 8642 | 8553 | 8464 | 8375 | 8286 | 8197 | 8108 | 8019 | 7929 | 7840 | 7775 | 7662 | 7573 | 7484 | 7395 | 7306 | 7217 | 7128 | 7038 | 6949 | 6860 | 6771 | 6682 | 6593 | 6504 | 6415 | 6326 |
| 28 | 88295 | 8741 | 8652 | 8564 | 8476 | 8388 | 8299 | 8211 | 8123 | 8034 | 7496 | 7858 | 7769 | 7681 | 7593 | 7505 | 7416 | 7328 | 7240 | 7151 | 7063 | 6975 | 6887 | 6798 | 6710 | 6622 | 6533 | 6445 | 6357 | 6268 |
| 29 | 87462 | 8659 | 8571 | 8483 | 8396 | 8308 | 8221 | 8133 | 8046 | 7959 | 7871 | 7784 | 7696 | 7609 | 7521 | 7434 | 7346 | 7259 | 7171 | 7084 | 6996 | 6909 | 6822 | 6734 | 6647 | 6559 | 6472 | 6384 | 6297 | 6209 |
| 30 | 86603 | 8573 | 8487 | 8400 | 8313 | 8227 | 8140 | 8054 | 7967 | 7880 | 7794 | 7707 | 7621 | 7534 | 7447 | 7361 | 7274 | 7188 | 7101 | 7014 | 6928 | 6841 | 6755 | 6668 | 6581 | 6495 | 6408 | 6322 | 6235 | 6148 |
| 31 | 85717 | 8485 | 8399 | 8313 | 8228 | 8142 | 8050 | 7970 | 7885 | 7799 | 7713 | 7628 | 7542 | 7456 | 7370 | 7285 | 7200 | 7114 | 7028 | 6943 | 6857 | 6771 | 6685 | 6600 | 6514 | 6428 | 6343 | 6257 | 6171 | 6085 |
| 32 | 84805 | 8395 | 8311 | 8226 | 8141 | 8056 | 7971 | 7886 | 7802 | 7717 | 7632 | 7547 | 7463 | 7378 | 7293 | 7208 | 7123 | 7038 | 6954 | 6869 | 6784 | 6699 | 6614 | 6529 | 6445 | 6360 | 6275 | 6190 | 6105 | 6021 |
| 33 | 83867 | 8303 | 8219 | 8135 | 8051 | 7967 | 7883 | 7799 | 7716 | 7632 | 7548 | 7464 | 7380 | 7296 | 7212 | 7128 | 7044 | 6960 | 6877 | 6793 | 6709 | 6625 | 6541 | 6457 | 6373 | 6290 | 6206 | 6122 | 6038 | 5954 |
| 34 | 82904 | 8207 | 8124 | 8041 | 7959 | 7876 | 7793 | 7710 | 7627 | 7544 | 7461 | 7378 | 7295 | 7212 | 7129 | 7047 | 6963 | 6881 | 6798 | 6715 | 6632 | 6549 | 6466 | 6383 | 6300 | 6218 | 6135 | 6052 | 5969 | 5886 |
| 35 | 81915 | 8109 | 8027 | 7945 | 7863 | 7782 | 7700 | 7618 | 7536 | 7454 | 7372 | 7290 | 7208 | 7126 | 7044 | 6962 | 6881 | 6799 | 6717 | 6635 | 6553 | 6471 | 6389 | 6307 | 6225 | 6143 | 6062 | 5980 | 5898 | 5816 |
| 36 | 80902 | 8009 | 7922 | 7847 | 7766 | 7685 | 7604 | 7523 | 7442 | 7362 | 7281 | 7200 | 7119 | 7038 | 6957 | 6876 | 6795 | 6715 | 6634 | 6553 | 6472 | 6391 | 6310 | 6229 | 6148 | 6067 | 5987 | 5906 | 5825 | 5744 |
| 37 | 79864 | 7906 | 7826 | 7747 | 7669 | 7587 | 7507 | 7427 | 7347 | 7267 | 7188 | 7107 | 7028 | 6948 | 6868 | 6788 | 6708 | 6628 | 6549 | 6465 | 6389 | 6309 | 6229 | 6149 | 6069 | 5989 | 5910 | 5830 | 5750 | 5670 |
| 38 | 78801 | 7801 | 7722 | 7643 | 7565 | 7486 | 7407 | 7328 | 7249 | 7170 | 7092 | 7013 | 6934 | 6855 | 6777 | 6698 | 6619 | 6540 | 6461 | 6382 | 6304 | 6225 | 6146 | 6067 | 5989 | 5910 | 5831 | 5752 | 5673 | 5995 |
| 39 | 77715 | 7694 | 7616 | 7538 | 7460 | 7382 | 7305 | 7227 | 7150 | 7072 | 6994 | 6916 | 6838 | 6761 | 6683 | 6605 | 6528 | 6450 | 6372 | 6294 | 6217 | 6139 | 6062 | 5984 | 5906 | 5828 | 5750 | 5673 | 5595 | 5517 |
| 40 | 76604 | 7583 | 7507 | 7430 | 7354 | 7277 | 7200 | 7124 | 7047 | 6970 | 6894 | 6817 | 6741 | 6664 | 6588 | 6511 | 6434 | 6358 | 6281 | 6204 | 6128 | 6051 | 5975 | 5898 | 5822 | 5745 | 5668 | 5592 | 5515 | 5438 |
| 41 | 75471 | 7471 | 7396 | 7320 | 7245 | 7169 | 7094 | 7018 | 6943 | 6867 | 6792 | 6716 | 6641 | 6565 | 6490 | 6415 | 6339 | 6264 | 6188 | 6113 | 6037 | 5962 | 5886 | 5811 | 5735 | 5660 | 5584 | 5509 | 5433 | 5358 |
| 42 | 74314 | 7357 | 7282 | 7208 | 7134 | 7059 | 6985 | 6911 | 6836 | 6762 | 6688 | 6613 | 6539 | 6465 | 6391 | 6316 | 6242 | 6168 | 6093 | 6019 | 5945 | 5870 | 5796 | 5722 | 5647 | 5573 | 5499 | 5424 | 5350 | 5276 |
| 43 | 73135 | 7240 | 7167 | 7094 | 7020 | 6947 | 6874 | 6801 | 6728 | 6655 | 6582 | 6509 | 6435 | 6362 | 6289 | 6216 | 6143 | 6070 | 5997 | 5923 | 5850 | 5777 | 5704 | 5631 | 5558 | 5485 | 5411 | 5338 | 5265 | 5192 |
| 44 | 71934 | 7121 | 7049 | 6977 | 6905 | 6833 | 6761 | 6689 | 6617 | 6545 | 6474 | 6402 | 6330 | 6258 | 6186 | 6114 | 6042 | 5970 | 5898 | 5826 | 5754 | 5682 | 5610 | 5538 | 5466 | 5395 | 5323 | 5251 | 5179 | 5107 |
| 45 | 70711 | 7000 | 6929 | 6858 | 6788 | 6717 | 6646 | 6576 | 6505 | 6434 | 6363 | 6293 | 6222 | 6151 | 6081 | 6010 | 5939 | 5869 | 5798 | 5727 | 5656 | 5586 | 5515 | 5444 | 5374 | 5303 | 5232 | 5161 | 5091 | 5020 |
| 46 | 69466 | 6877 | 6807 | 6738 | 6668 | 6599 | 6529 | 6460 | 6390 | 6321 | 6251 | 6182 | 6113 | 6043 | 5974 | 5904 | 5835 | 5765 | 5696 | 5626 | 5557 | 5487 | 5418 | 5388 | 5279 | 5209 | 5140 | 5071 | 5001 | 4932 |
| 47 | 68200 | 6751 | 6683 | 6615 | 6547 | 6479 | 6410 | 6342 | 6274 | 6206 | 6138 | 6069 | 6001 | 5933 | 5865 | 5797 | 5728 | 5660 | 5592 | 5524 | 5456 | 5387 | 5319 | 5251 | 5183 | 5115 | 5046 | 4978 | 4910 | 4842 |
| 48 | 66913 | 6624 | 6557 | 6490 | 6423 | 6356 | 6289 | 6222 | 6155 | 6089 | 6022 | 5955 | 5888 | 5821 | 5754 | 5687 | 5620 | 5553 | 5486 | 5419 | 5353 | 5286 | 5219 | 5152 | 5085 | 5018 | 4951 | 4884 | 4817 | 4750 |
| 49 | 65606 | 6494 | 6429 | 6363 | 6298 | 6232 | 6166 | 6101 | 6035 | 5970 | 5904 | 5838 | 5773 | 5707 | 5642 | 5576 | 5510 | 5445 | 5379 | 5314 | 5248 | 5182 | 5117 | 5051 | 4986 | 4920 | 4854 | 4789 | 4723 | 4658 |
| 50 | 64279 | 6363 | 6299 | 6235 | 6170 | 6106 | 6042 | 5977 | 5913 | 5849 | 5785 | 5720 | 5656 | 5592 | 5527 | 5463 | 5399 | 5335 | 5270 | 5206 | 5142 | 5078 | 5013 | 4940 | 4885 | 4820 | 4756 | 4692 | 4628 | 4563 |
| 51 | 62932 | 6230 | 6167 | 6104 | 6041 | 5978 | 5915 | 5852 | 5789 | 5726 | 5663 | 5600 | 5538 | 5475 | 5412 | 5349 | 5286 | 5223 | 5160 | 5097 | 5034 | 4971 | 4908 | 4845 | 4782 | 4719 | 4656 | 4594 | 4531 | 4468 |
| 52 | 61566 | 6095 | 6033 | 5971 | 5910 | 5848 | 5787 | 5725 | 5664 | 5602 | 5540 | 5479 | 5417 | 5356 | 5294 | 5233 | 5171 | 5109 | 5048 | 4986 | 4925 | 4863 | 4802 | 4740 | 4679 | 4617 | 4555 | 4494 | 4432 | 4371 |
| 53 | 60182 | 5958 | 5897 | 5837 | 5777 | 5717 | 5657 | 5596 | 5536 | 5476 | 5416 | 5356 | 5296 | 5235 | 5175 | 5115 | 5055 | 4995 | 4934 | 4874 | 4814 | 4754 | 4694 | 4634 | 4573 | 4513 | 4453 | 4393 | 4333 | 4272 |
| 54 | 58779 | 5819 | 5760 | 5701 | 5642 | 5584 | 5527 | 5466 | 5407 | 5348 | 5290 | 5231 | 5172 | 5113 | 5054 | 4996 | 4937 | 4878 | 4819 | 4761 | 4702 | 4643 | 4584 | 4525 | 4467 | 4408 | 4349 | 4290 | 4232 | 4173 |
| 55 | 57358 | 5678 | 5621 | 5563 | 5506 | 5449 | 5391 | 5334 | 5276 | 5219 | 5162 | 5104 | 5047 | 4990 | 4932 | 4875 | 4818 | 4760 | 4703 | 4645 | 4588 | 4531 | 4473 | 4416 | 4359 | 4301 | 4244 | 4187 | 4129 | 4072 |
| 56 | 55919 | 5535 | 5480 | 5424 | 5368 | 5312 | 5256 | 5200 | 5144 | 5088 | 5032 | 4976 | 4920 | 4864 | 4809 | 4753 | 4697 | 4641 | 4585 | 4529 | 4473 | 4417 | 4361 | 4305 | 4249 | 4193 | 4138 | 4082 | 4026 | 3970 |
| 57 | 54464 | 5391 | 5337 | 5283 | 5228 | 5174 | 5119 | 5065 | 5010 | 4956 | 4901 | 4847 | 4792 | 4738 | 4683 | 4629 | 4574 | 4520 | 4466 | 4411 | 4357 | 4302 | 4248 | 4193 | 4139 | 4084 | 4030 | 3975 | 3921 | 3866 |
| 58 | 52992 | 5246 | 5193 | 5140 | 5087 | 5034 | 4981 | 4928 | 4875 | 4822 | 4769 | 4716 | 4663 | 4610 | 4557 | 4504 | 4451 | 4398 | 4345 | 4292 | 4239 | 4186 | 4133 | 4080 | 4027 | 3974 | 3921 | 3868 | 3815 | 3762 |
| 59 | 51504 | 5098 | 5047 | 4995 | 4944 | 4892 | 4841 | 4789 | 4738 | 4686 | 4635 | 4583 | 4532 | 4480 | 4429 | 4377 | 4326 | 4274 | 4223 | 4171 | 4120 | 4068 | 4017 | 3965 | 3914 | 3862 | 3811 | 3795 | 3708 | 3656 |
| 60 | 50000 | 4950 | 4900 | 4850 | 4800 | 4750 | 4700 | 4650 | 4600 | 4550 | 4500 | 4450 | 4400 | 4350 | 4300 | 4250 | 4200 | 4150 | 4100 | 4050 | 4000 | 3950 | 3900 | 3850 | 3800 | 3750 | 3700 | 3650 | 3600 | 3550 |

If a mistake has been made in measuring background, then the factor for normalizing the coefficients to give $A_0 = 1$ will be wrong. In this case, no value of $D$ or $\varepsilon$ will satisfy the expression for $A_n$ as a function of $n$. However, this is easily detected by comparing the $A_n$'s with the table for several values of $n$ and, if necessary, a more accurate normalizing constant could be chosen. In order to demonstrate that the determination of the background level was no problem in measurements taken on electroplated films, theoretical curves are computed and indicated as small circles, together with experimentally determined functions of $A_n$, in Fig. 1. The agreement between the theoretical points and the experimental curves is quite within the limits of applicability.

### Application to measurements on powders and films

Using annealed compacts of electrolytic iron powders, measurements of $\varepsilon$ and $D$ computed with this technique were compared with values computed using the standard procedure with several profiles. The values for $\varepsilon$ and $D$ computed with the two techniques agreed within significant limits.

Figure 1 illustrates the type of problem in electroplated films for which this technique is applicable. The plotted curves show the influence of saccharin and $p$H on the strained crystallite size and are representative of a large number of curves recorded in this study.[4] Each curve shown is the plot of measurements taken on two separate films plated under identical conditions (six films altogether). Each set of films represents a different plating condition of $p$H and saccharin content in the plating bath. The computed values of $\varepsilon$ and $D$ are listed as follows:

| | $p$H | $\varepsilon$ | $D$ |
|---|---|---|---|
| Plated without saccharin | 2.1 | 0.0055 | 210 A |
| Plated without saccharin | 3.1 | 0.0045 | 595 A |
| Plated with saccharin, 840 gms/$l$ | 3.1 | 0.0037 | 895 A |

A detailed analysis of structure is an extremely difficult task using presently developed technology. For example, lattice strain distributions and crystallite size distributions are reasonably expected to influence profile shapes. Consequently, the techniques described above can be expected to give only a qualitative picture of the strain and of the average crystallite size. Nevertheless, the parameters $\varepsilon$ and $D$ computed from (3) have proved to be very useful in correlating observed physical properties with electrodeposition parameters.

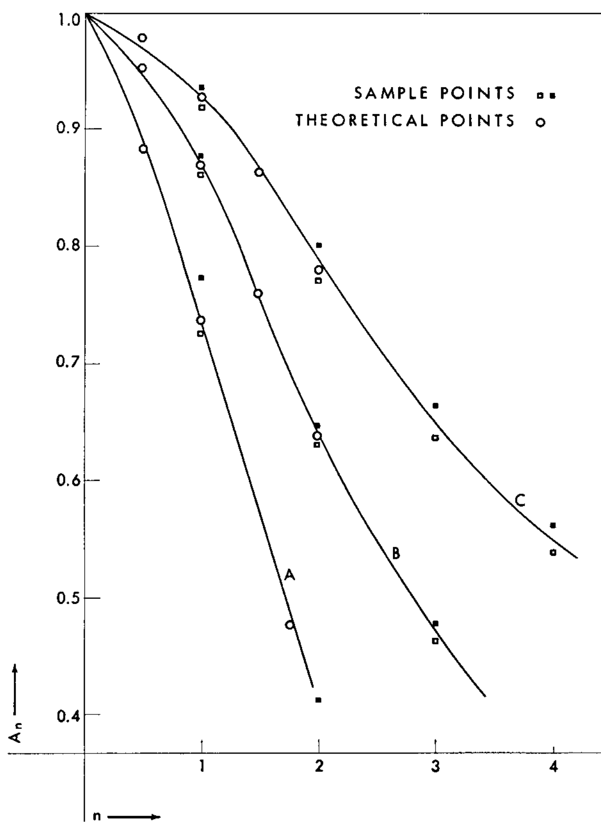


*Figure 1* **Diffraction profiles of electroplated films.** *Parameter $A_n$ computed for* FeNi *electroplated films with varying* $p$H *and saccharin content of the plating bath. Open and solid squares are computed points for the two specimens.*

A. *Saccharin deficient,* $p$H = 2.1

B. *Saccharin content 420 mg/L,* $p$H = 3.1

C. *Saccharin deficient,* $p$H = 3.1

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