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MAGNETIC METHOD FOR MEASURING THE THICKNESS OF NICKEL COATINGS ON NONMAGNETIC BASE **METALS**

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ABSTRACT

The local thickness of an electroplated nickel coating on a nonmagnetic base metal may be measured by the force of attraction of a small permanent magnet, the attractive force being determined with a simple spring balance. It was found that the attractive force between the magnet and coating is proportional to the thickness of the coating. The instrument is calibrated with nickel coatings of known thickness which have been deposited under about the same conditions of known thickness which have been deposited under about the same conditions as the coatings to be tested. Nickel coatings deposited under different conditions have somewhat different magnetic properties, which become uniform by heating the samples to 400° C (750° F). Specimens of unknown history should be annealed before testing. Measurements on coatings as deposited are correct within ± 15 percent and on annealed coatings within ± 10 percent. The effect on magnetic measurements of factors such as curvature of the

surface, type of base metal, and superposed chromium coatings is discussed. The magnetic method was shown to be applicable to the testing of nickelplated brass plumbing fixtures.

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I. INTRODUCTION

Until recently, the thickness of electroplated coatings was usually specified in terms of the average thickness, which may be computed from the weight of coating found, for example, by chemical analysis, on a measured area. However, many specifications now refer to the minimum thickness on significant surfaces, for the determination of which it is necessary to measure the local thickness at one or more points.

For the latter purpose the most generally applicable procedure is the metallographic method, in which the thickness of a suitably prepared cross section is measured by means of a microscope and scale. This method requires experience and expensive equipment and results in destruction of the specimen tested.

The "dropping" method ¹ for zinc and cadmium deposits, and the very similar jet method 2 for nickel coatings, depend on the time required for a specified reagent to dissolve the coating. The "chord" method ³ involves the width of the cut made when the coating on a flat surface is just penetrated by a grinding wheel of known radius, or on a curved surface by a flat grinding tool. These methods destroy the coatings but have little effect on the base metal, which can usually be refinished.

Especially for factory inspection, there is a great need for a nondestructive test for thickness, which, if conditions warrant, may be applied to the entire product of a plant. The present paper describes a nondestructive method for measuring the thickness of nickel coatings on nonmagnetic base metals.

II. PRINCIPLE

The proposed method depends on the force of attraction between a permanent magnet and the nickel coating. It is entirely distinct from those methods⁴ in which the thickness of a nonmagnetic coating (which may be nonmetallic) upon steel or iron is measured in terms of the reluctance of a magnetic circuit which passes through a coil and through the steel under the coating. In effect, this latter method measures the gap between the magnet and the base metal, and its use is confined to nonmagnetic coatings on steel having uniform thickness and magnetic properties.

The same principle is involved in a method ⁵ in which the thickness of a nonmagnetic coating on steel is measured in terms of the force required to detach a magnetic needle from the surface.

It is well known that nickel is ferromagnetic and has a permeability about one-fourth that of iron. In the present work it was found that the attractive forces between a permanent magnet and thin layers of nickel, having the same magnetic permeability, are proportional within about 10 percent to the thickness of the layers, for nickel coatings from 0.0005 mm (0.00002 in.) to 0.025 mm (0.001 in.), which is the

¹ S. G. Clarke, J. Electrodepositors' Tech. Soc. 8, Paper no. 11 (May 1933); and R. O. Hull and P. W. C. Strausser, Monthly Rev. Am. Electroplaters' Soc. 22 (March 1935).
² S. G. Clarke, J. Electrodepositors' Tech. Soc. 12, preprint (1936).
³ W. Blum and A. Brenner. Mesle's chord method for measuring the thickness of metal coatings. J. Research NBS 16, 171 (1936) RP 866.
⁴ Electric Thickness Gage, General Electric Co. Instruments 8, 341 (1935). A similar device was demonstrated by Octavius Knight at a conference on electroplating in New York on September 26, 1934.
⁵ L. V. Radtchenko and F. K. Shestakovsky, J. Tech. Phys. (USSR), 5, 1372 (1935).



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FIGURE 1.-de Noüy tensiometer used as a magnetic balance.

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FIGURE 2.—Sanford magnetic balance with cover removed.

range of thickness of practically all commercial nickel coatings upon nonferrous metals. For thicker coatings an empirical curve may be constructed.

In principle, the proposed method involves merely the measurement of the force required to detach one pole of a permanent magnet from the nickel coating, and the comparison of this force with that required to detach the same magnet from a similar nickel coating of known thickness.

Nickel coatings deposited under different conditions have somewhat different magnetic permeabilities. It was found that if such coatings are annealed at 400° C (750° F) they acquire about the same permeability. The magnetic method is therefore more reliable for annealed coatings than for coatings as deposited.

III. METHOD OF MEASUREMENT

1. TYPES OF BALANCES

In order to determine the force of attraction between the magnet and the coating, any convenient device may be used that will measure forces of 1 to 5 g with a precision of about 1 percent. As the instrument must be calibrated empirically, it is necessary to know only the relative (and not the actual) loads, for example, in terms of proportional scale divisions. Three types of balances were employed in this investigation, namely, lever, torsion, and spring balances. In their design and operation, an effort was made to define conditions that will yield a precision of at least 5 percent, which is adequate for commercial testing of nickel coatings. Unless otherwise stated, all conclusions regarding favorable conditions or limitations will be based upon that criterion.

(a) LEVER BALANCE

An analytical balance was used in preliminary measurements to determine the validity and sensitivity of the method. The magnet was suspended and balanced from one arm so that it could be brought into contact with the nickel surface. Weights were then added to the other arm until the magnet was just detached. In one test a cylindrical bar magnet made of 36-percent cobalt steel, and 15 cm (6 in.) long and 5 mm (0.2 in.) in diameter, with a hemispherical end, was attracted to a nickel coating 0.025 mm (0.001 in.) thick with a force of about 2.5 g. This load could be measured readily to within 1 percent.

Thickness tests with such a balance are not convenient, especially for coatings on articles of irregular shape. This method is therefore not recommended except for orienting experiments.

(b) TORSION BALANCE

A du Noüy tensiometer for measuring surface tension can be readily adapted to the desired purpose. As shown in figure 1, a small bar magnet 2 mm (0.08 in.) in diameter and 3.0 cm (1.2 in.) long is suspended from the lever arm in place of the platinum loop that is used in surface-tension measurements. The torsion in the wire is adjusted so that with the freely suspended magnet a zero reading is obtained. The nickel surface to be tested is then raised until it is just in contact

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with the magnet, and the force necessary to detach the magnet is measured on the scale.

When equipped with a freely suspended magnet, this instrument yields slightly more reproducible results than does either of the spring balances to be described. It is, however, not so compact or convenient as the latter, and the zero point requires more frequent adjustment.

(c) SPRING BALANCE

The simplest form of spring balance consists merely of a vertical helical spring, from the lower end of which a small magnet is suspended. The force necessary to detach the magnet from a nickel coating is measured by the extension of the spring. A few tests with such a device showed that it is usable but inconvenient and requires frequent adjustments.

A compact, portable spring balance, shown in figure 2, was designed a few years ago by R. L. Sanford ⁶ for measuring the magnetic permeability of austenitic steel. One of these instruments, which will be referred to as the "Sanford balance", was used in making most of the thickness measurements referred to in this paper. In this device two small bar magnets, each 5 mm (0.2 in.) in diameter and 5 cm (2 in.) long, are arranged astatically (to eliminate the effect of the earth's magnetic field) and are pivoted upon a sensitive jeweled bearing. The force required to detach the end of one magnet from a magnetic surface is measured by means of a spring and dial.

The sensitivity of this instrument can be varied by proper selection of the spring. With a suitable spring it was found that each division on the dial corresponded to approximately 0.00025 mm (0.00001 in.) of nickel, so that the 100 divisions covered the range of thickness usually applied to nonferrous metals. The reproducibility was about ± 3 percent on coatings down to about 0.005 mm (0.0002 in.).

The Sanford balance was found to be satisfactory upon plane and most convex surfaces but not upon concave surfaces, because the magnet does not extend below the base of the instrument. The "modified Sanford" balance shown in figures 3 and 4 was designed to overcome this limitation. By removing the false bottom, the end of the magnet can be applied to a concave surface. The two small magnets, arranged astatically, are vertical, that is, normal to the surface tested, as then a more nearly uniform flux is produced about the end of the magnet than exists with an inclined magnet. The two magnets are carried on a duralumin lever arm, which reduces the weight of the moving parts and increases the sensitivity. The rack and pinion facilitate the application of the magnet to large or irregularly shaped articles. Both forms of Sanford balance are applicable in any position, which is an advantage in testing large articles or those in fixed positions.

When testing with a magnetic balance, it is expedient to make a rapid preliminary measurement in order to get an approximate reading, which is then approached more slowly in the subsequent test. The plated surface should be clean. Lack of duplication of readings is usually caused by grease or other foreign material on the surface.

⁶ R. L. Sanford, A magnetic balance for the inspection of austenitic steel. BS J. Research 10, 321 (1933) RP532.

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FIGURE 3.—Modified Sanford magnetic balance.

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FIGURE 4.—Modified balance with cover and false-bottom removed.

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2. POSITION OF MAGNET AND DIRECTION OF APPLIED FORCE

The maximum force of attraction between a magnet and a nickel coating is exerted in a direction normal to the surfaces at the point of contact. If a magnet is pulled from a surface in a normal direction, the detaching force is equal to the attractive force. However, if the detaching force is not applied along the normal, it measures only a component of the attractive force, and the detaching force is proportional to the cosine of the angle between its direction and the normal to the surface. This relation was confirmed experimentally. The angle of inclination of the bar magnet to the normal is not important; only the direction of the detaching force enters into consideration.

When using a freely suspended magnet on a lever balance or tensiometer, the surface to be measured should be set normal to the magnet. In this case, the detaching force is equal to the attractive force. A departure of 10° from the normal reduces the detaching force by only 2 percent.

With a balance having a magnet rigidly attached to a lever arm, the direction of the detaching force is along the tangent to the circle described by the end of the magnet and having the pivot as the center. With both the Sanford balance and the modified instrument, the detaching force is about 75 percent of the attractive force. When making measurements, the angle between the normal to the surface and the direction of the detaching force should always be the same. This condition is met by having the plated surface normal to the magnet of the modified balance, or parallel to the base of the original Sanford balance. For a precision of 5 percent, the angle between the magnet and the normal to the surface should vary by not more than 5° . Less variation can be permitted in this angle than with a freely suspended magnet, as the cosine changes more rapidly in the neighborhood of 40° than near 0°. When either type of Sanford balance is set on a plane surface, the magnets will contact the surface at the proper angle. On curved surfaces, the magnet of the modified balance can be set normal to the surface, by eye, within about 5°. When testing a cylindrical surface it is easier to secure the proper position if the axis of the lever which carries the magnets is placed parallel to the axis of the cylinder, rather than perpendicular to it.

3. SIZE AND COMPOSITION OF THE MAGNET

The magnet may be of any composition that is suitable for permanent magnets. Most of the experiments here reported were made with magnets of 36-percent cobalt steel suitably heat treated. A few tests with Alnico magnets showed that they have somewhat more attractive force for a given weight, and depart less from linearity between attractive force and thickness of nickel coating than do the cobalt-steel magnets. Their principal disadvantage is that they cannot be machined, but must be ground to the desired size and shape. The strength of new magnets of either type tends to decrease during the first few weeks, after which, however, they require calibration only at long intervals. They should not be brought into contact with other magnets or with strongly magnetic metals.

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In general, small magnets are preferable to large ones because a more sensitive balance may be used, they are more suitable for measuring small or curved surfaces, and they are less affected by a nickel coating on the opposite side of a plated sheet. On the other hand, the attractive force of magnets 2 mm (0.08 in.) or less in diameter shows larger departures from linearity with respect to thickness, especially of thin coatings, and hence must be calibrated for a greater number of thicknesses. Taking all of these factors into account, an Alnico magnet 2 mm (0.08 in.) in diameter and 3 cm (1.2 in.) long was selected for the instrument shown in figure 4. The contact end of the magnet is hemispherical.

4. STANDARDIZATION OF APPARATUS

(a) PREPARATION OF THICKNESS STANDARDS

It is necessary to calibrate the apparatus with nickel deposits of known thickness and of the same magnetic permeability as those to be tested subsequently. The preparation of deposits of uniform, accurately known thickness upon flat plates involves many difficulties.

For most of the tests, the nickel coating was deposited on one side of a flat plate, 10 by 15 cm (4 by 6 in.), the other side of which was insulated with a suitable stop-off lacquer. The specimens were mounted in a metal rack so that there was less than 6 mm (0.25 in.) either between adjacent specimens or between specimens and the rack, and the plating was done in a rectangular tank of which the rack and specimens completely filled the cross section.

The average thickness, computed from the total weight of nickel deposited on a plate, was the basis of most of the magnetic measurements. On such specimens, the thickness of coating near the edges was from 10 to 20 percent above the average, but on 70 percent of the area the thickness was uniform and not more than 4 percent below the average thickness computed from the total weight of deposit. The average thickness over the 70 percent that constituted the central portion was computed more accurately by cutting off strips about 1 cm (0.4 in.) wide from each edge, determining the weight of nickel on them, and deducting this amount from the total weight of the original deposit. The magnitude of this correction was from 3 to 5 percent. Magnetic and metallographic measurements at numerous points on this central portion showed that the mean deviation from the average thickness was about ± 4 percent, and the maximum deviation was about ± 8 percent.

On many specimens, the thickness of deposit was confirmed by microscopic measurements of metallographic sections at the same points that were measured magnetically.

For measuring the effects of curvature upon magnetic tests, cylinders and spheres were plated uniformly by rotating them during the plating process.

The solutions and conditions used in plating are summarized in table 1.

Magnetic Thickness Measurements

002			Conc	entrati	on	Usual conditions						
Bath	Туре	Composition	N	g/liter	oz/gal	pH	Tepera	em- ature	Current	density		
E'	Standard	NiSO4.7H2O NiCl2.6H2O H3BO3	1.4 0.4 .5 M	$200 \\ 45 \\ 30$	27 6 4	5.3	°C 35	°F 95	amp/dm ₂	amp/ft ² 19		
F	Low pH	NiSO4.7H2O NiCl2.6H2O H3BO3	1.4 0.4 .5 M	$200 \\ 45 \\ 30$	27 6 4	2.9	35	95	2	19		
G	High sulphate.	NiSO4.7H2O NH4Cl Na2SO4 H3BO3	.55 .3 1.6 0.25 M	$77 \\ 15 \\ 120 \\ 15 \\ 15$	10 2 16 2	5.7	27	81	1	9		
н	KCla	NiSO4.7H2O KCl H3BO3	1.7 0.25 .5 M	240 19 30	32 2.5 4	3.0	35	95	1.5	14		
I	Double salt	NiSO4(NH4)2SO4.6H2O _ NH4Cl H3BO3	$^{4}_{.3}_{.25 M}$	75 15 15	10 2 2	5.3	30	86	0.3	28		

TABLE 1.—Conditions used in nickel plating

^a Bath used by G. E. Gardam and D. J. Macnaughtan, Trans. Faraday Soc. 29, 756 (1933), and designated as "NSB.KC".

(B) CALIBRATION

If a linear relation exists, the calibration of a given magnet and instrument may be conveniently expressed in terms of the equation

C = T/F

where

C = a constant.

T=the thickness of the nickel deposit, expressed in fractions of a millimeter or an inch.

F=the attractive force (or more strictly, the detaching force), expressed in grams or in scale divisions.

In effect, C is the thickness of nickel corresponding to 1 g or one scale division. As most commercial specifications for thickness are expressed in fractions of an inch, usually less than 0.001 inch, C may be conveniently expressed as hundred thousandths of an inch per scale division. If, for example,

T/F=0.00030/20=0.000015, then $C=1.5\times10^{-5}$ inch, or conventionally, C=1.5.

In calibrating a magnet or instrument it is preferable to employ several similar standards of known thicknesses, in order to obtain average values of C that apply to that type of coating. The constancy of C for similar nickel deposits of different thicknesses indicates that a linear relation exists between thickness and magnetic attraction. Similarly, the constancy of C for dissimilar nickel coatings of the same thickness is a measure of the constancy of magnetic properties of different nickel deposits. In general, an increase in the value of C represents a decrease in the magnetic permeability of the nickel, and vice versa.

(1)

IV. MAGNETIC PROPERTIES OF NICKEL DEPOSITS

1. RELATION BETWEEN MAGNETIC ATTRACTION AND THICKNESS

Table 2 summarizes the relation of thickness to magnetic attraction, expressed in terms of C for coatings as deposited and of C' for coatings that were annealed, for the reason above mentioned. The data in tables 2 and 3 were obtained with the Sanford balance. The actual values of the constants C and C' apply only to this particular instrument and are useful only for comparison. Other magnets would give parallel results, although the values of C and C' might differ from those of the above instrument. Each value of C or C' in the tables is the average of 10 to 20 measurements on one or more specimens.

For any given type of nickel deposit, it is evident that the value of C is constant to within about 10 percent for thicknesses from 0.0001 to 0.001 in. (0.0025 to 0.025 mm). A few results not included in table 2 show that for much thinner coatings, such as 0.00002 to 0.00005 in. (0.0005 to 0.0013 mm), the value of C is about 20 percent below the average in the table.

The average value of C varies, however, by about 20 percent for different types of nickel deposits, which must, therefore, have different magnetic permeabilities.

TABLE 2.—Ratio between thickness of nickel (T) and magnetic attraction (F)

				Avera	ige thi	icknes	s of n	ickel ((inch)				1.20		Ava
Bath	Base metal	10×10-5		20×	20×10-5		30×10-5		50×10-5		(10-5	Avg.	dev.	Avg.	dev.
	t i seni i seti	C	<i>C</i> ′	C	<i>C</i> ′	C	C'	C	<i>C</i> ′	C	<i>C</i> ′	C	%	<i>C</i> ′	%
E	Low brass	{1.66	1.06	1. 91	1. 13	1. 88	1. 14	1. 74	1.12	1. 49	1.09	1. 74	±8	1. 11	±3
F	Low brass	{1.75	1.12	1. 84	1. 14	1.84	1. 11	1. 70	1.09	1.82	1.07	1. 79	± 3	1. 11	± 2
G	Low brass	{2.00	1. 22	2. 21	1.26			2. 27	1, 22	2. 21	1.20	2. 17	± 4	1. 23	±2
G	Zine	1.63		1.78		1.71		1. 93		1.68		1.75	± 5		
G	$ \{ \begin{matrix} {\rm Zinc} & {\rm die\text{-}cast} \\ {\rm XXI} \end{matrix} $	{2.16	1. 29			2. 26	1. 27	2. 11	1.25	2.08	1. 20	2.15	±3	1.25	±3
G	Zinc die-cast XXIII	{2.02		2. 24	1. 26	2.13		2. 13	1, 22	2.14	1.14	2. 13	±3	1. 21	±3
Η	Low brass	{		2.18	1. 28			2.08	1.19			2. 13	±3	1.24	±4
I	Low brass	{		2.03	1.24			2. 24	1.17			2.14	± 5	1. 20	±3
Avera	ge	1.87	1.17	2.03	1.22	1.96	1.17	2.03	1.18	1.90	1.14	1. 98		1.19	
Avera	ge deviation for d	ifferen	nt bat	hs (%)							±10		±5	

C=T/F for coatings as deposited; C'=T/F after annealing ¹ (Flat plates. Sanford balance) In this table the values of C and C' are to be multiplied by 10⁻⁵

 1 Coatings on brass were heated to 400° C (750° F) for 30 minutes; on zinc and zinc-base die-castings to 280° C (535° F) for 60 minutes.

Brenner]

Magnetic Thickness Measurements

TABLE 3.—Effect of operating conditions on the magnetic constants of nickel

[Values of C and C' derived from coatings 0.013 mm (0.0005 in.) thick on flat-rolled brass. Sanford balance]

Exper- iment	pН	Current	density	Tem	ipera- ire	C	Devia- tion from avg C	C'	Devia- tion from avg C'	Variable
Sec.		ginds	a gant	1.1	ST	ANDARD	BATH	E		Index Managers
1 2 3	5.0 5.0 5.0	$\frac{\operatorname{amp}/\operatorname{dm}^2}{\mathop{1}\limits_{\begin{array}{c}2\\4\end{array}}}$	amp/ft ² 9 19 37	°C 35 35 35	°F 95 95 95	1.76×10 ⁻⁵ 1.74 1.77	$\begin{vmatrix} \frac{\%}{-7} \\ -8 \\ -6 \end{vmatrix}$	$\begin{vmatrix} 1.12 \times 10^{-5} \\ 1.12 \\ 1.16 \end{vmatrix}$		Current density.
4 5 6 7	5.4 5.4 5.4 5.4 5.4	2 2 2 2 2	19 19 19 19	$21 \\ 35 \\ 45 \\ 60$	70 95 113 140	$\begin{array}{c} 2.20 \\ 1.91 \\ 2.01 \\ (2.54) \end{array}$	$ \begin{array}{c} +16 \\ +1 \\ +6 \\ (+34) \end{array} $	1.191.231.241.22	$^{+1}_{+4}_{+5}_{+3}$	}Temperature.
8 9 10 11 12 13 14 15 16	$\begin{array}{c} 2.7\\ 2.9\\ 4.4\\ 5.2\\ 5.6\\ 5.8\\ 5.8\\ 5.9\end{array}$	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	19 19 19 19 19 19 19 19 19	35 35 35 35 35 35 35 35 35 35	95 95 95 95 95 95 95 95 95	$\begin{array}{c} 1.70\\ 1.80\\ 2.03\\ 1.76\\ 1.80\\ 2.00\\ (2.7)\\ (3.3)\\ (5.6) \end{array}$	$ \begin{array}{c} -10 \\ -5 \\ +7 \\ -7 \\ -5 \\ +6 \\ (+40) \\ (+70) \\ (+190) \end{array} $	$\begin{array}{c} 1.\ 09\\ 1.\ 11\\ 1.\ 23\\ 1.\ 10\\ 1.\ 11\\ 1.\ 18\\ 1.\ 18\\ 1.\ 19\\ 1.\ 25\\ \end{array}$	-8 -6 +4 -7 -6 0 0 +1 +6)рн.
17 18 19	5.8 5.8 5.8	$\begin{array}{c}1\\2\\4\end{array}$	9 19 37	35 35 35	95 95 95	(3.2) (3.3) (4.4)	(+70) (+70) (+130)	$1.17 \\ 1.19 \\ 1.22$	$^{-1}_{+1}_{+3}$	Current density at high pH.
20 21 22	$5.2 \\ 5.2 \\ 5.2 \\ 5.2$	$\begin{array}{c}2\\2\\2\end{array}$	19 19 19	35 35 35	95 95 95	2.06 (5.4) (7.6)	+9 (+180) (+300)	1.21 (1.33) (1.84)	$^{+3}_{(+13)}_{(+57)}$	$2 \text{ ml/liter of } 3\% \text{ H}_2\text{O}_2.$ 5 ml/liter of $3\% \text{ H}_2\text{O}_2.$ 10 ml/liter of $3\% \text{ H}_2\text{O}_2.$
A	verag these	e, excludin s	ng values	in pa	aren-	1.89	±7	1. 18	±4	
		e isist.	1973 - 945	I	IIGH	-SULPHA'	ГЕ ВАТ	TH G		singres de s
23 24	6.1 6.1	$\begin{vmatrix} 1\\2 \end{vmatrix}$	9 19	35 35	95 95	2.08 2.14	$-{2 \atop 0}$	1.18 1.29	$^{-1}_{+8}$	}Current density.
25 26 27	$\begin{array}{c} 6.1 \\ 6.1 \\ 6.1 \end{array}$	1 1 1	9 9 9	27 33 48	81 91 118	2.31 2.08 1.78	$^{+8}_{-2}_{-16}$	1.17 1.18 1.19	$-2 \\ -1 \\ 0$	}Temperature.
28 29 30 31 32	$\begin{array}{r} 4.5 \\ 5.7 \\ 6.1 \\ 6.4 \\ 6.6 \end{array}$	1 1 1 1 1	9 9 9 9	27 27 27 27 27 27	81 81 81 81 81 81	$\begin{array}{c} 2.\ 28\\ 2.\ 21\\ 2.\ 31\\ 1.\ 93\\ 2.\ 20 \end{array}$	+7 +4 +8 -9 +3	$\begin{array}{c} 1.21\\ 1.23\\ 1.17\\ 1.17\\ 1.13 \end{array}$	+2 +3 -2 -2 -5	}pH.
A	ver- age_					2.13	± 6	1.19	± 3	a stagener (1
33 34						1.38 1.17		1.24 1.11		Bright nickel. ¹ Bright nickel. ¹

¹Deposits in experiments 33 and 34 are from different commercial baths.

As shown in table 2, suitable annealing of the various nickel coatings causes them to acquire about the same magnetic properties, that is, the average values of C' (for annealed coatings) are more nearly uniform than the values of C (for unannealed coatings). Moreover, the linear relation was more closely followed by the annealed coatings, for which C' is constant within 5 percent, for the entire range of thickness from 0.0005 to 0.025 mm (0.00002 to 0.001 in.). This linear relationship was found to hold also for nickel coatings on curved surfaces.

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These results were obtained with the Sanford balance, which has magnets 5 cm (2 in.) in length and 5 mm (0.2 in.) in diameter. For smaller magnets the linear relation does not hold quite so well for coatings thinner than about 0.0075 mm (0.0003 in.). For example, the modified Sanford balance with magnets 3 cm (1.2 in.) in length and 2 mm (0.08 in.) in diameter showed a departure of about 10 percent from linearity for thin annealed coatings. Therefore, small magnets should be calibrated against at least three thicknesses of nickel coatings, for example, 0.013 mm (0.0005 in.), 0.0075 mm (0.0003 in.), and 0.0025 mm (0.0001 in.).

2. EFFECTS OF CONDITIONS OF DEPOSITION

(A) NICKEL AS DEPOSITED

Table 3 summarizes more extensive experiments in which the plating conditions were systematically varied. The results show that for deposits plated out of the standard bath, the value of C is fairly constant over a range of conditions that includes most commercial plating. The value of C is increased, that is, the nickel is made less magnetically permeable, by an increase in temperature above 45° C (113° F), or by an increase in pH above 5.5 (experiments 7 and 14). At a high pH, an increase in current density (experiment 19) still further increases C, but not at a low pH (experiment 3). The amount of hydrogen peroxide usually added to reduce pitting (experiment 20) has no marked effect on C, but additions of more than 2 ml/liter (0.26 fl oz/gal) of 3-percent peroxide (experiments 21 and 22) have a marked effect on C.

For deposits plated out of the high-sulphate bath, the average value of C is somewhat higher than in the standard bath, but an increase in pH (experiments 28 to 32) has much less effect in the high-sulphate than in the standard bath.

The conclusion from these tests on coatings as deposited is that the magnetic method may be applied to unannealed coatings only if the conditions of operation are maintained fairly uniform. Under such conditions, it is merely necessary to calibrate the instrument at intervals against deposits of known or measured thickness that were produced in the same bath and under the prescribed conditions. With these precautions, the method may be conveniently applied in factory production without introducing errors greater than about 15 percent.

The direct method is not, however, satisfactory for inspecting coatings of unknown origin that may have been plated under varied conditions. For such testing, prior annealing of the deposits is necessary.

(B) ANNEALED COATINGS

The lower and more constant values of C' in tables 2 and 3 for annealed coatings show that heating nickel deposits to 400° C (750° F) for 15 to 30 minutes makes them more magnetic and at the same time more nearly alike in magnetic permeability. The use of a single average constant for the principal types of nickel deposits yields results that are correct within about 10 percent. The only exceptions are experiments 21 and 22, in which an excess of hydrogen peroxide was used.

Annealing of nickel coatings in air at 400° C (750° F) produces a yellowish discoloration of oxide which, however, does not interfere

Brenner]

with magnetic measurements. It can be removed with a warm cyanide solution. Annealing can be done more quickly at still higher temperatures, but then there is increased oxidation of the nickel surface. When the usual thin layer of chromium is present over the nickel, no appreciable change in color or luster is produced on annealing at 400° C, and the chromium has no effect on the magnetic readings.

The values of C' in table 2 for zinc-base die-castings were determined after annealing at 280° C (535° F) for 1 hour. This is about the highest temperature that can be used on such alloys without damaging the coatings, and even at that temperature there is softening and distortion of the castings. The magnetic method is not nondestructive for such articles if annealing is required.

No consistent results could be obtained on nickel-plated zinc annealed at 280° C. Apparently the coating and base metal had alloyed. It is surprising that the die-castings that contained over 90 percent of zinc showed no such alloying at that temperature.

(C) "BRIGHT NICKEL" DEPOSITS

No attempt was made to study exhaustively the behavior of bright nickel deposits, such as are now being produced from numerous patented or proprietary baths. Results on deposits made commercially from two different baths that contained organic addition agents are reported in experiments 33 and 34 of table 3. These data show that, as deposited, these coatings yielded lower values of C, that is, were much more magnetic than ordinary nickel. On annealing they changed but little, and the values of C' approached closely to the average value of C' for other nickel deposits. These few tests indicate that it may be possible to test bright nickel directly if the instrument has been calibrated against similar deposits; but that it would be safer to anneal the coatings and use the average value of C'

(D) EFFECTS OF CODEPOSITED IRON OR COBALT

As both iron and cobalt are more magnetic than nickel, their presence in a nickel deposit would be expected to yield low values of both C and C'. The results in table 4 confirm this prediction. The deposits containing iron were prepared from regular nickel baths to which ferrous sulphate was added. The ratio of iron to nickel in the deposits was about twice that in the baths. The single nickel-cobalt alloy that was tested was prepared commercially from a bright nickel bath containing cobalt and an organic addition agent.

The production of the second second		Co	c			Annealed			
Thickness	Fe			in th caused	ge of error ickness by 1% of	<i>C</i> '	Percentage of error in thickness caused by 1% of		
di w moniegos i			10 to get 1	Fe	Co	210.20	Fe	Co	
Inch 50×10 ⁻⁵	%	%	1.85×10-5	%	%	1.17×10-5	%	%	
25 65 25 55	3.5 3.5 11 11 11		1.77 1.50 1.24 1.24	$ \begin{array}{r} 1.3 \\ 6.6 \\ 4.5 \\ 4.5 \\ 4.5 \end{array} $		1.03 1.02 0.80 .78	3.9 4.2 4.3 4.5		
30		20	1.09		3.5	. 83		2.0	
A vorage				4 2	3.5		4.2	2.0	

TABLE 4.—Effect of iron and cobalt on magnetic thickness measurements

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These few results indicate that in either annealed or unannealed coatings the presence of each percent of iron causes an error of about +4 percent, and in annealed coatings each percent of cobalt an error of about +2 percent in the magnetic readings. With the present general use of anodes containing more than 99 percent of nickel, coatings will not contain over 1 percent of iron or cobalt and hence introduce significant errors. If cobalt is intentionally introduced, as in the bright deposit tested, the magnetic method is applicable only if the cobalt content is kept fairly constant, and if the instrument is standardized against similar coatings.

3. RELATION OF COMPOSITION, HARDNESS, AND MAGNETIC PROPERTIES

In order to throw light on the variations in magnetic properties, a few corresponding, but thicker, nickel deposits were analyzed ⁷ for oxygen and hydrogen by vacuum fusion. The results in table 5 show that in the standard bath an increase in pH raised the oxygen

Experi- ment	Deth	- 11	Oxygen	Hydro-	Brinell	Magnetic	c constant	Waniahla
	вати	рн	(% by wt)	by wt)	ness no.	C	C'	Variable
1 2 3	E E G	5. 2 5. 8 6. 6	0.004 .021 .014	0.0017 .0060 .0053	120 269 470	1.7×10 ^{−5} 2.7 2.2	1. 10×10 ⁻⁵ 1. 18 1. 13	Standard. Standard, high pH. High sulphate, high pH.

TABLE 5.—Relation between gas content and properties of electrodeposited nickel

content of the deposit more than fivefold. In the high-sulphate bath (experiment 3) at a high pH, there was almost as much oxygen as in experiment 2. These amounts of oxygen correspond to about 3 times as much nickel hydroxide, Ni(OH)₂, in which form the oxygen is most likely present. The contents of hydrogen were roughly proportional to those of oxygen, but in all cases far exceeded the amounts of hydrogen needed to combine with the oxygen in Ni(OH)₂ or H_2O . The contents of hydrogen and oxygen are in the same order as the values of C for the unannealed deposits, that is, a high value of C (low magnetic permeability) corresponds to a high content of oxygen and hydrogen. The fact that in experiment 2 the deposit is harder than in experiment 1 is consistent with the hypothesis of Macnaughtan and his associates 8 that hardness is caused by, or associated with, included basic material. However, as deposit 3, with less oxygen, is harder than no. 2, it is evident that other factors, such as grain size, may also influence the hardness.

If the basic material is concentrated on the grain boundaries, it may reduce the magnetic permeability either by hindering magnetic coupling of the grains, or by producing internal strains, which are known to reduce the permeability. The increase in permeability by heating to as low a temperature as 400° C is more consistent with the removal of strains than with a reaction with, or absorption of, the oxide. Analysis of an annealed specimen of nickel showed that the annealing considerably reduced the content of hydrogen but did not change that of oxygen. It is evident that the subject warrants more exhaustive study.

 ⁷ Analyses were made by V. Holm.
 ⁸ D. J. Macnaughtan, G. E. Gardam, and R. A. J. Hammond, Trans. Faraday Soc. 29, 729 (1933).

4. EFFECT OF BASE METAL

The method is necessarily limited to nonmagnetic base metals. None of the common nonferrous metals tested was magnetic enough to interfere with the application of this method. Even nickel-brass, which contains about 20 percent of nickel, is nonmagnetic, and hence nickel coatings over nickel-brass can be measured magnetically. However, when testing coatings on unknown alloys, it is desirable to apply the magnet to a portion of the unplated or stripped surface. If only a small magnetic attraction is observed, this may be deducted from the readings made on the coatings. Any large magnetic effect makes the method inapplicable to such base metals.

Nickel coatings were plated on a number of different base metals for the purpose of determining the effect of the base metal on the magnetic properties of the coating. For nickel coatings plated on copper, various kinds of rolled, extruded, and cast brass, rolled nickel-brass, and zinc-base die castings, the values of C and C'showed no effect of the base metal greater than 5 percent. For unannealed coatings on sheet zinc the value of C was 15 percent lower than on the other base metals.

V. DIMENSIONAL FACTORS IN MAGNETIC THICKNESS MEASUREMENTS

1. AREA OF COATING

Only a limited portion of the nickel surface exerts an appreciable effect upon the magnetic attraction. To determine the magnitudes of the effective areas, the attraction of a given sized magnet was measured, first to a relatively large plane surface of nickel, which for present purposes may be considered as an "infinite plane", and then to successively smaller disks of the same plated sheet.

TABLE 6.—Effect of area of nickel coating on attractive force of typical magnets.

Expressed as percentage of the attraction to an infinite plane. Deposits about 0.013 mm (0.0005 in.) thick

Ex- peri- ment				м	agnet		Percentage of attraction of disks of the following diameters:						
	Diamete		Length		Material	F	F/W^{a}	Balance	4 cm (1.6 in.)	2 cm (0.8 in.)	1 cm (0.4 in.)	0.75 cm (0.3 in.)	0.5 cm (0.2 in.)
1 3 b 4 5 6 7 8 9 10	cm 1.4 .5 .2 .5 .2 .2 .2 .2 .2 .2 .2 .1 .1	in. 0.56 .2 .08 .20 .08 .08 .08 .08 .08 .04 .04	cm 12 15 15 5 3 3 2.4 .7 6.0 3.0	in. 5 6 2 1.2 1.2 1.0 0.3 2.4 1.2	36% of Co 36% of Co 36% of Co 36% of Co 7001 steel Alnico 36% of Co 36% of Co	g 4.8 2.4 2.8 1.7 1.1 0.5 1.2 .9 .7 .6	$\begin{array}{c} 0.03 \\ .1 \\ .2 \\ 1.5 \\ .6 \\ 2.1 \\ 6.0 \\ 1.8 \\ 3.8 \end{array}$	Lever do Sanford Lever du Noüy do do do	$\begin{array}{c} Per-\\cent \\ 84\\100\\100\\100\\100\\100\\100\\100\\100\\100\\10$	Per- cent 56 93 94 97 100 100 100 100 100 100	Per- cent 17 73 79 77 90 89 90 90 90 97 94 96	Per- cent 10 64 73 66 87 87 88 94 94 95	Per- cent 7 48 66 52 78 77 78 87 90 90

F = Force of attraction (in grams) between the magnet and a nickel coating 0.025 mm (0.001 in.) thick.

W = Weight of magnet (in grams). • The magnet and a noise total group of 0.5 cm was reduced to a diameter of 0.2 cm (0.08 in.)

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The results in table 6 show that the smaller the diameter of the magnet the smaller is the area over which the magnet exerts 90 percent of its attraction to an infinite plane.

As a first approximation, the diameter of the disk that is effective is about 5 times the diameter of the magnet. Reliable measurements upon small areas therefore require the use of small magnets, preferably not more than about 2 mm (0.08 in.) in diameter.

The area influenced by the magnet is even more significant in making thickness measurements near an edge or corner. These measurements should be made with the magnet placed at such a distance from the edge or corner as to include in its field a disk of the diameter that was found by tests such as those in table 6 to yield 90 percent or more of the attractive force. For most small magnets this distance is about twice the diameter of the magnet.

With either form of the Sanford balance, consistent measurements can be made nearer to an edge than indicated above if the lever arm of the balance extends over the surface of the coating and not over the edge. Reliable measurements can then be obtained at a distance from the edge equal to about one-half the diameter of the magnet.

2. CURVATURE OF SURFACE

The tests thus far described were made on flat surfaces. To test the effects of curvature, brass cylinders and spheres were plated uniformly with nickel. The magnetic attractions of the coatings were measured and were expressed in terms of the attraction to an infinite plane having the same thickness of nickel.

TABLE 7.—Effect of curvature expressed as percentage of the attraction to an infinite plane

				Percentage attraction of magnets of following diameters-					
Experiment	Shape	Rad	lius	0.5 cm 0.2 in.	^a 0.2 cm 0.08 in.	0.2 cm 0.08 in.	0.1 cm 0.04 in.		
1 2 34	Spheredo	cm 1 0.6 .3 .16	in. 0.4 .25 .13 .06	% 93 84 65 37	% 94 89 78 51	% 93 88 88 88 65	% 93 91 97 78		
5 6 7 8	Cylinder do dodo	1.40.6.3.16	.6 .25 .13 .06	100 101 93 75	102 100 94 83	100 100 94 86	100 100 98 88		

Deposits about 0.013 mm (0.0005 in.) thick

• See table 6, magnet 3.

The results in table 7 show that, in order to yield about 95 percent of the maximum attractive force, a sphere should have a radius at least 5 times the diameter of the magnet, and a cylinder should have a radius at least twice the diameter of the magnet. Smaller spheres or cylinders can be measured if an empirical correction is made.

No quantitative tests were made upon concave surfaces, but their geometry indicates that a decrease in the radius of curvature would increase the magnetic attraction above that on a plane surface. As a first approximation it may be assumed that the limitations on the radius of curvature will be the same as on convex surfaces.

3. PARALLEL NICKEL COATINGS

The above measurements were all made on specimens plated with nickel on only one side. Any nickel coating that is present on the back of a sheet will exert an effect upon the measurement on the front, that depends largely upon the thickness of the intervening base metal. This effect is illustrated in figure 5, in which are plotted the proportions of the usual attractive forces of these particular magnets that were exerted by coatings separated from the magnet by various distances.



FIGURE 5.—Decrease in magnetic attraction with distance of magnet from coating (Expressed as percentage of attractive force when magnet and coating are in contact)

Curve		Ma	Deposit				
	Dian	neter	Len	igth			
A B C D	mm 5 5 2 2	in. 0.2 .2 .08 .08	mm 150 150 30 30	in. 6 1.2 1.2	As deposited. Annealed. As deposited. Annealed.		

These separations were obtained by placing a brass sheet of the designated thickness between the magnet and the coating. The attractive force varies inversely as the square of the distance from the effective pole of the magnet. As, however, this pole does not coincide with the end of the magnet, from which point the distances were measured, the inverse square law is not followed by these curves. The curves show that the attractive force falls off more rapidly with increase in distance of the less permeable, unannealed coatings (A and C) than of the annealed coatings (B and D). Figure 5 and additional data show that with unannealed coatings the thickness of sheet must be at least 1.5 times the diameter of the magnet in order to reduce the effect of a coating on the far side to 5 percent or less, and with annealed coatings at least 3 times the diameter of the magnet. It is evident, therefore, that the magnetic method cannot generally be used directly to measure the thickness of nickel on one side of sheet metal that has nickel on the other side. To test such specimens, the nickel coating on the opposite side of the sheet may be removed by grinding or by swabbing with nitric acid.

Such curves as those in figure 5 may be used for computing approximately the thickness of nickel on each side of a sheet from magnetic measurements on each side. The attractive force of a magnet for two thin parallel layers of nickel is equal to the sum of their separate attractions in the same positions.

Let C=the magnetic constant for that instrument and type of nickel coating.

 F_1 =the attraction when the magnet is in contact with coating 1.

 F_2 = the attraction in contact with coating 2.

- d= the distance between the coatings, i. e., the thickness of the base metal sheet.
- p=the fraction of the contact attraction that is obtained at a distance d. (p is read from curves such as those in fig. 5).

 f_1 = the attraction that would be caused by coating 1 alone, when in contact with the magnet.

 f_2 = the attraction that would be caused by coating 2 alone, when in contact with the magnet.

Then,

$$F_1 = f_1 + p f_2, \tag{2}$$

$$F_2 = pf_1 + f_2, \tag{3}$$

$$f_1 = \frac{F_1 - pF_2}{1 - p^2} \tag{4}$$

$$f_2 = \frac{F_2 - pF_1}{1 - p^2} \tag{5}$$

$$f(\text{avg.}) = \frac{f_1 + f_2}{2} = \frac{F_1 + F_2}{2(1+p)} \cdot$$
(6)

The corresponding thicknesses are obtained by

$$T_1 = C f_1, \tag{7}$$

$$\Gamma_2 = Ct_2. \tag{8}$$

$$T(avg_{i}) = Cf(avg_{i}), \tag{9}$$

A few measurements of coatings of known thickness on both sides of a sheet yielded correct results within about 5 percent for each side and for the average, provided the two thicknesses were about the same. If, however, one coating is several times as thick as the other, large proportional errors are made in computing the thickness of the thin coating. The above method is therefore only an approximation, but it may be useful in testing a given fairly uniform product.

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4. SUPERPOSED CHROMIUM COATINGS

As previously noted, the presence of chromium over the nickel is an advantage if the coatings are to be annealed, as it prevents oxidation. The curves of figure 5 and other data show that decorative chromium coatings, which are usually less than 0.0013 mm (0.00005 in.) thick, have very little effect on the attractive force of the underlying nickel. Tests may therefore be made directly on the chromiumplated surfaces.

VI. APPLICABILITY TO COMMERCIAL TESTING

The accuracy of the method was finally checked by comparing the magnetic measurements with the thicknesses measured metallographically at approximately the same points. The results in table 8 show that on pieces plated uniformly or otherwise, the average error was about ± 5 percent, which compares favorably with the accuracy of metallographic measurements of thin coatings and meets most commercial requirements. The systematic errors obtained with a given specimen show that the magnetic constant of the coating differed slightly from the average value of C obtained by calibration.

The accuracy of routine measurements will depend largely upon the uniformity of the magnetic properties of the coatings. Especially if these are tested without annealing, it is desirable to check the magnet calibration at intervals against annealed deposits or by metallographic measurements of specimens of similar shape. The latter precaution will minimize any errors due to size or curvature.

	Article		Local thinic	ickness of kel	Error	Average		
Experiment	Shape	Size, di	ameter	Metallo- graphic	Magnetic	Error	error	
1	Cylinder (annealed)	cm 2.8	in. 1.1	$ \begin{bmatrix} \text{in.} \\ 39 \times 10^{-5} \\ 38 \\ 42 \\ 42 \\ 42 \end{bmatrix} $	$ \begin{array}{c} \text{in.} \\ 40 \times 10^{-5} \\ 39 \\ 41 \\ 42 \end{array} $		<i>%</i> } +1	
2	Cylinder (not annealed)	5	2	$\left\{ \begin{array}{c} 56 \\ 58 \\ 59 \\ 56 \end{array} \right.$	60 59 61 60	+7 +2 +3 +3 +7	} +5	
3	Cylinder (annealed)	5.8	2.3	$\left(\begin{array}{c} 91\\ 90\\ 80\\ 70\\ 67\\ 65\\ 70\\ 74\end{array}\right)$	99 101 85 74 68 68 68 71 81		$\left.\right\rangle$ +6	
4	Flat (annealed)	10×15	4×6	$\left\{ \begin{array}{c} 63 \\ 44 \\ 36 \\ 37 \\ 37 \\ 44 \end{array} \right.$	63 42 34 31 33 46	$ \begin{array}{c} 0 \\ -5 \\ -6 \\ -16 \\ -11 \\ +5 \end{array} $	} -6	
le hearachti	Average error						±5	

TABLE 8.—Comparison of magnetic and metallographic measurements

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The approximate accuracy of the method for miscellaneous plated pieces was confirmed by making exploratory magnetic measurements upon about 50 plated brass plumbing fixtures that were subsequently stripped to determine their compliance with Federal specifications for average thickness. The coatings were annealed at 400° C prior to the magnetic testing, and a single magnetic constant was applied.

The average of the magnetic measurements on each piece agreed within 15 percent with the average thickness found by stripping, on almost all of the pieces that approached the specified minimum average of 0.00018 in. (0.0045 mm). This agreement is good, in view of the fact that there is no practical way to "weight" the magnetic readings with respect to corresponding areas, as is done effectively by stripping. The magnetic method is not therefore a convenient or very accurate method for measuring average thickness. It is designed chiefly for measuring the local thickness and distribution.

The results with the above fixtures throw light on the actual distribution of nickel on typical articles, such as plumbing fixtures. The average ratio of the maximum to the minimum thickness was about 2.5:1, but on some pieces this ratio was as high as 5:1. If a minimum thickness, e. g., of 0.0001 inch (0.0025 mm) of nickel were specified on such articles, it is apparent that on some pieces an average thickness of at least 0.0003 inch would have to be applied.

VII. CONCLUSIONS

1. The attractions of a small permanent magnet for nickel coatings on nonmagnetic base metals are proportional to their thicknesses, up to about 0.025 mm (0.001 inch); and can, therefore, be used to measure the thickness at any point.

2. This magnetic attraction can be measured by any fairly sensitive balance, for example, (a) a lever balance, such as is used for chemical analysis, (b) a torsion balance, such as a du Noüy tensiometer, or (c) a spring balance, such as that devised by R. L. Sanford for magnetic measurements.

3. The magnetic permeabilities of nickel deposits depend to some extent on the conditions of deposition. In general, deposits produced at a high pH, or from baths that yield hard or fine-grained deposits, are less permeable magnetically than soft deposits. The so-called "bright nickel" coatings, from baths with organic addition agents, are, however, more permeable than normal deposits.

4. Nickel deposited at a high pH is harder and contains more oxygen and hydrogen than that at a low pH.

5. Heating at 400° C (750° F) for 15 to 30 minutes causes nearly all nickel deposits to acquire about the same magnetic permeability.

6. The magnetic method can be quickly applied to nickel coatings deposited on nonferrous metals under fairly uniform conditions, provided that the instrument has been calibrated against similar nickel coatings of known thickness.

7. If nickel coatings of unknown history have been annealed at 400° C (750° F), their thickness may be measured magnetically, provided the constant for annealed coatings is used.

8. Each percent of iron in a nickel deposit introduces an error of about +4 percent in the thickness measurements, and each percent of cobalt an error of about +2 percent.

9. On plane surfaces the magnetic measurement yields correct results if the diameter of the area tested is at least 5 times the diameter of the magnet.

10. Measurements closer to an edge or corner than about twice the diameter of the magnet are inaccurate unless the magnet has been especially calibrated for such positions.

11. On curved surfaces the results are practically the same as on planes, provided the radius of curvature of a sphere is at least 5 times the diameter of the magnet and the radius of a cylinder twice the diameter of the magnet.

12. The measurements on sheet metal are affected by the presence of a nickel coating on the other side unless the thickness of the base metal is at least 3 times the diameter of the magnet. It is possible to compute the approximate thickness on each side of a thin sheet from magnetic measurements made on both sides.

13. The presence of the usual thin decorative chromium coatings over the nickel has no appreciable effect on the thickness measurements, and is advantageous because it prevents oxidation of the nickel during annealing.

14. The magnetic method is rapid and nondestructive, and for thin coatings its accuracy approaches that of metallographic measurements.

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