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Methods of thickness test for metallic coatings

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at the Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently **JIS H 8501:1988** is replaced with **JIS H 8501:1999**.

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Methods of thickness test for metallic coatings

Introduction This Japanese Industrial Standard has been prepared based on the corresponding International Standards listed in Remarks to 1 **Scope**, without any modification in technical contents, and the items not specified in the corresponding International Standards are complementarily added as a part of the Japanese Industrial Standard.

1 Scope This Japanese Industrial Standard specifies the methods of thickness test for the electric and chemical metallic coatings on the basis metal.

Remarks: The International Standards corresponding to this Standard are as follows.

- ISO 1463 : 1982 Metallic and oxide coatings — Measurement of coating thickness — Microscopical method
- ISO 2064 : 1996 Metallic and other inorganic coatings — Definitions and conventions concerning the measurement of the thickness
- ISO 2177 : 1985 Metallic coatings — Measurement of coating thickness — Coulometric method by anodic dissolution
- ISO 2178 : 1982 Non-magnetic coatings on magnetic substrates — Measurement of coating thickness — Magnetic method
- ISO 2360 : 1982 Non-conductive coatings on non-magnetic basis metals — Measurement of coating thickness — Eddy current method
- ISO 3497 : 1990 Metallic coatings — Measurement of coating thickness — X-ray spectrometric methods
- ISO 3543 : 1981 Metallic and non-metallic coatings — Measurement of thickness — Beta backscatter Method
- ISO 3868 : 1976 Metallic and other non-organic coatings — Measurement of coating thickness — Fizeau multiple-beam interferometry method
- ISO 3882 : 1986 Metallic and other non-organic coatings — Review of methods of measurement of thickness
- ISO 4518 : 1980 Metallic coatings — Measurement of coating thickness — Profilometric method
- ISO 9220 : 1988 Metallic coatings — Measurement of coating thickness — Scanning electron microscope method

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. The most recent editions of the standards indicated below shall be applied.

JIS B 0601 *Surface roughness — Definitions and designation*

JIS B 7503 *Dial gauges*

JIS B 7519 *Microindicators*

JIS B 7520 *Indicating micrometers*

JIS B 7533 *Dial test indicators (lever type)*

JIS B 7536 *Electrical comparators*

JIS H 0400 *Glossary of terms used in electroplating and related processes*

JIS K 0132 *General rules for scanning electron microscopy*

3 Definitions For the main terms used in this Standard the definitions in **JIS H 0400** apply, and the rest of the terms are as follows.

- a) **significant surface** The part of the article covered or to be covered by the coating and for which the coating is essential for serviceability and/or appearance and where the coating must meet all of the specified requirements.
- b) **measuring area** The area of the significant surface over which a single measurement is made.
- c) **reference area** The area within which a specified number of single measurements is required to be made.
- d) **local thickness** The mean of the thickness measurements, of which a specified number is made within a reference area.
- e) **minimum local thickness** The lowest value of the local thickness ensured on the significant surface of a single article.
- f) **maximum local thickness** The highest value of the local thickness ensured on the significant surface of a single article.
- g) **average thickness** Either the value obtained by analytical methods or the mean of a specified number of local thickness measurements that are evenly distributed over the significant surface.

4 Classes of test methods The classes of the test methods shall be as follows.

- a) Microscopic method for cross-sectional thickness test
- b) Coulometric method (by anodic dissolution) for thickness test
- c) Eddy current method for thickness test
- d) Magnetic method for thickness test
- e) X-ray spectrometric method for thickness test
- f) Beta back-scatter method for thickness test
- g) Multiple-beam interference test method
- h) Scanning electron microscope test method
- i) Micrometric method for thickness test
- j) Mass instrumentation method for deposit quantity test

5 Sample

5.1 Handling of sample Samples shall be handled not with bare hand, but with gloved hands.

5.2 Sampling Samples shall be taken from the significant surfaces of products or be products themselves. However, where measurement on products or referee is impossible, samples shall be substituted.

The samples shall be representative⁽¹⁾⁽²⁾ of products.

Notes ⁽¹⁾ The composition of the basis materials, the manufacturing condition and the finished condition before the coating should preferably be the same as those of the products.

⁽²⁾ The pre-treatment and the coating shall be achieved in the same bath and under the same conditions as those for the product, and should be achieved simultaneously with the product so that the effect of the working condition is reflected on the sample.

5.3 Size of sample The size of the samples shall be subjected to the agreement between the parties concerned with delivery.

5.4 Treatment of test surface The test surface shall be treated using an appropriate solvent ⁽³⁾ depending on the stain.

Note ⁽³⁾ For the solvents to be used ethyl-alcohol, benzine, volatile oil, etc. are preferable.

5.5 Conditioning of sample The samples shall be generally left in a room or an air-conditioned room at $(23 \pm 2)^\circ\text{C}$ and not more than 65 % in relative humidity for conditioning before the test is started. But the conditioning may be omitted subject to the agreement between the parties concerned with delivery when it is not inconvenient for the test.

6 General conditions of test room

6.1 Test place The test place shall be generally in the room at $(23 \pm 2)^\circ\text{C}$ and not more than 65 % in relative humidity. The test may be carried out at an appropriate place under the agreement between the parties concerned with delivery when there is no inconvenience for the test.

6.2 Maintenance of test apparatus The test apparatus shall be correctly installed on a firm test table, and stabilized so as not to generate any abnormal motion accompanied by the test. The instruments to be used shall be maintained so as to be constantly used under the specified condition.

7 Determination of local thickness

7.1 Product with significant surface less than 100 mm² When the significant surface of the product is less than 100 mm², the whole significant surface of the product shall be the reference area to be used for determining the local thickness. The number of measurements within the reference area shall be subjected to the agreement between the parties concerned with delivery.

Remarks: In a special case, the reference area may be reduced, but the size, number and the position shall be subjected to the agreement between the parties concerned with delivery.

7.2 Product with significant surface not less than 100 mm² When the significant surface of the product is not less than 100 mm², the reference area of about 100 mm² (preferably a square of 1 cm in side length) of the significant surface of the product shall be the reference area to be used for determining the local thickness. The measurement shall be made at maximum 5 points within this reference area dispersedly.

- Remarks
- 1 The number of measurements and the positions within the reference area shall be subjected to the agreement between the parties concerned with delivery.
 - 2 In the measurement by the microscopic method for cross-sectional thickness test, the measurement shall be made at least 5 points in the length of the designated section dispersedly.

8 Determination of average thickness

8.1 Mass instrumentation method When the average thickness is determined by the mass instrumentation method, the area of measurement shall be determined so that the mass reduction which is enough to ensure the accuracy can be obtained in the weighing method to be used.

- Remarks
- 1 When the significant surface of the product is less than the minimum area required for the measurement, the minimum area shall be ensured by increasing the number of the products, and the obtained result of measurement shall be the average thickness.
 - 2 When the significant surface of the product does not greatly exceed the minimum area required for the measurement, the obtained result of measurement shall be the average thickness.
 - 3 When the significant surface of the product markedly exceeds the minimum area required for the measurement, the measurements of the designated number shall be dispersedly made on the significant surface, and the result shall be individually indicated.

8.2 Other methods

- a) When the significant surface of the product does not greatly exceed the reference area required for determining the local thickness, the obtained local thickness shall be the average thickness.
- b) When the significant surface of the product markedly exceeds the reference area required for determining the local thickness, three to five times of measurements shall be dispersedly made on the significant surface, and the obtained average shall be the average thickness.

9 Microscopic method for cross-sectional thickness test

9.1 Summary This is a method to measure the thickness of coating by a microscopic observation of the vertical cross-section of coating.

9.2 Apparatus The apparatus with ocular scale inserted or a micrometer microscope shall be used. An example of apparatus is shown in Fig. 1.

Remarks: This figure is not intended to show the standard for the shape and structure.

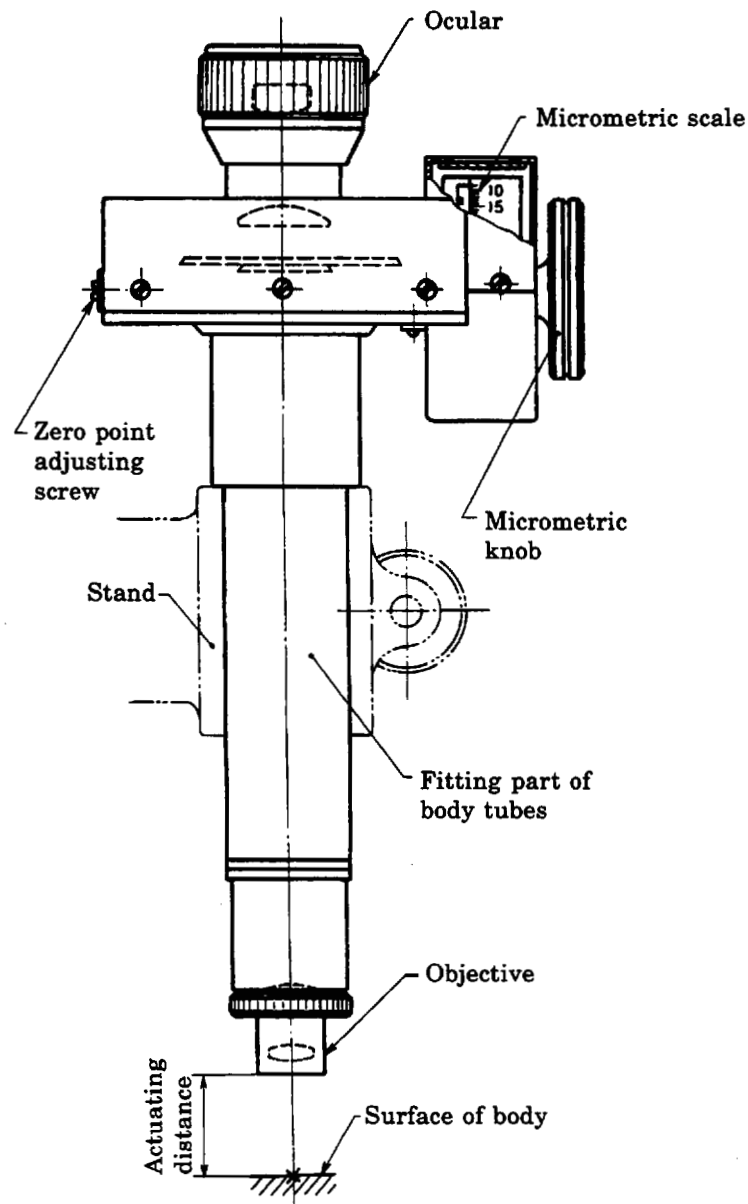


Fig. 1 Example of microscope

9.3 Operation Mount the test piece⁽⁴⁾ in the synthetic resin or the alloy having low melting point so that the coated surface is vertical, and polish⁽⁵⁾ it in the perpendicular direction to the coated surface with care not to damage the coating. Apply corrosive treatment to the basis material by using corrosive liquid given in Table 1 so that the boundary between the basis material and the coating can be distinguished, magnify it appropriately using a micrometer microscope, measure the thickness of coating. Fig. 2 shows an example of reading method by a movable marked line.

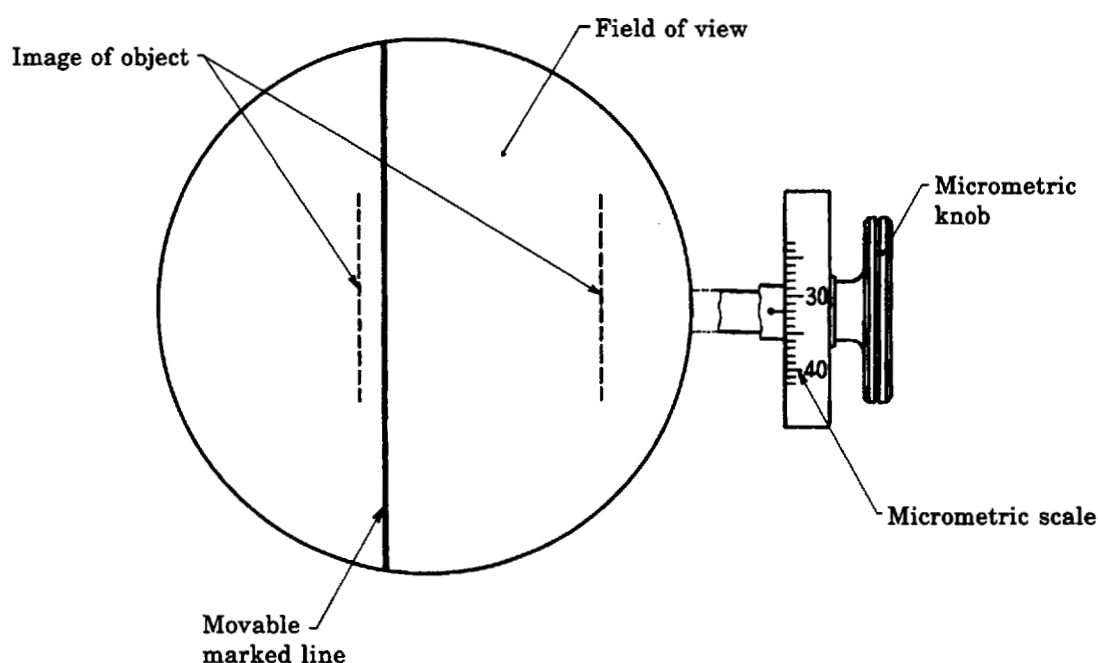
Table 2 shows the relation between the thickness of coating and magnification.

Notes ⁽⁴⁾ For a soft coating of gold, silver, tin, etc., additionally apply a hard coating, such as nickel coating, of not less than 10 μm to prevent sags due to polishing.

- (⁵) Polishing shall be done to make the cross-section of the test piece clear. Polishing shall be conducted from a hard metal toward a soft metal to prevent sags where a test piece is placed perpendicularly to a polishing paper. The working angle of the test piece on the polishing paper shall be 45°, the test piece shall be turned by 90° whenever the polishing paper is exchanged, and polishing shall be made until traces caused by the previous polishing paper disappear.

Table 1 Examples of corrosive liquids

Application	Composition of corrosive liquid
Coating of nickel or chromium on iron and steel (Corrosive liquid for iron and steel)	Nitric acid (specific gravity 1.38) 5 ml Ethyl alcohol (95 wt %) 95 ml
Coatings of gold, lead, silver, nickel or copper on iron and steel, copper and its alloys (Corrosive liquid for iron and steel, copper and their alloys)	Ferric (II) chloride (hexahydrate) 10 ml Hydrochloric acid 2 ml (specific gravity 1.16) Ethyl alcohol (95 wt %) 98 ml
Multilayer deposits of nickel on iron and steel and copper alloys (Where the layer of nickel is classified such as mat, semi-bright and bright) (Corrosive liquid for nickel)	Nitric acid 50 ml (specific gravity 1.38) Glacial acetic acid 50 ml
Coating of tin or its alloys on copper and its alloys (Corrosive liquid for copper and its alloys)	Ammonium sulfate 10 g Ammonium hydroxide 2 ml (specific gravity 0.88) Pure water 90 ml
Coating of nickel or copper on aluminum and its alloys (Corrosive liquid for aluminum and its alloys)	Nitric acid 5 ml (specific gravity 1.38) Hydrofluoric acid 2 ml (specific gravity 1.14) Pure water 93 ml
Coating of nickel or copper on zinc alloys Coating of zinc or cadmium on iron and steel (Corrosive liquid for zinc, its alloys and cadmium)	Chromic anhydride 20 g Sodium sulfate 1.5 g Pure water 100 ml



- Remarks 1 The figure is not intended to show the standard for the shape and structure.
- 2 In measurement, move the marked line from the top of the image of object to its end using the micrometric knob and read the distance by the micrometric scale to determine the thickness.

Fig. 2 Example of reading method by movable marked line

Table 2 Relation between coating thickness and magnification

Coating thickness μm	Magnification
20 to 30	200
10 to 20	200 or 400
6 to 10	400

10 Coulometric method for thickness test

10.1 Summary This is a method to measure the thickness of coating by practical application of the fact that the time required for dissolving anodically and removing an extremely small area of coating by constant current electrolysis is proportional to the thickness of coating.

Informative reference: In the coulometric method for thickness test, the thickness of each film can be measured without changing the place of measurement for other multi-layered coating than a single-layered coating. In such a case, the film thickness shall be obtained by completely dissolving the coating of the upper layer, obtaining the thickness, removing the used electrolytic solution, effecting the sufficiently cleaning, placing the electrolytic solution obtained from

the relationship between the coating and the basis metal in the cell, and performing the operation similar to that for the coating of the upper layer. The thickness of the multi-layered film shall be successively measured by the similar operation.

10.2 Apparatus The apparatus shall consist of a constant electric current generating apparatus (rectifier), electrolytic tank and their accessories. Fig. 3 shows an example of this apparatus.

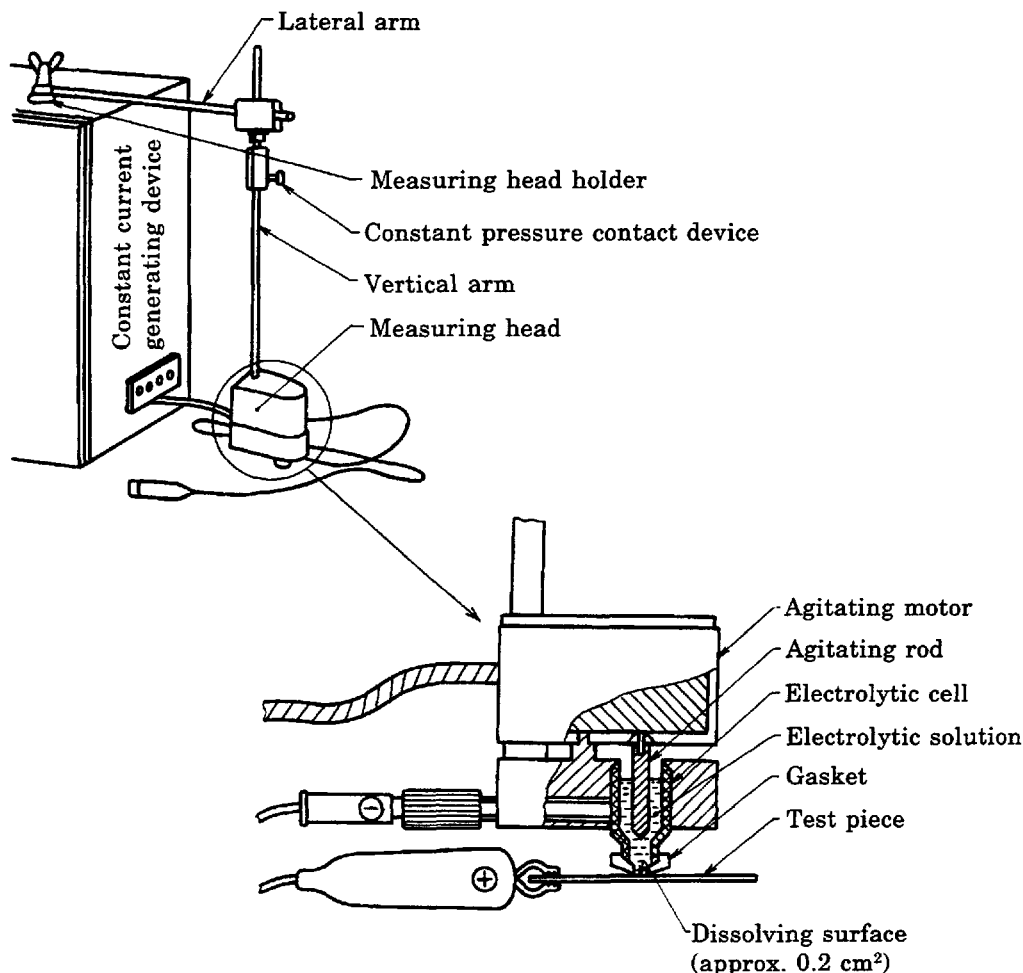


Fig. 3 Example of coulometric measuring apparatus

10.3 Calibration Sufficient calibration shall be conducted before use of this apparatus on the test. For the following items, care shall be taken at the time of calibration:

- The apparatus shall be calibrated according to the characteristics of apparatus by using a suitable standard samples before use of the apparatus. The calibrations shall also be conducted at appropriate intervals of time during the measurement.
- The standard samples to be used for calibration shall be uniform in thickness and be covered with the same class of coating of a known thickness.
- When calibrating, the qualities of basis material and coating of the calibration standard shall be the same or similar to the basis material and film of the sample to be measured.

10.4 Operation The operation shall be as follows:

- a) The apparatus shall be operated as indicated in the instructions for each apparatus.
- b) Examine whether the combination of basis material (or substrate), coating and electrolytic solution ⁽⁶⁾(⁷) is in conformity to Table 3 and Table 4.
- c) When the basis material has been laid bare as the coating is dissolved, the electric potential (or voltage) shows a rapid fluctuation. This point is the time of completion of electrolysis. Record the time required or the integrated quantity of electricity.
- d) If the coating of the part surrounded by the gaskets has been completely dissolved and removed after the test, the operation shall be completed.
- e) The thickness of coating shall be obtained in accordance with the instruction for the apparatus.

Note ⁽⁶⁾ The electrolytic solution used shall be such that it does not impair the coating when the operation specified in 10.4 is not being conducted, the anode current efficiency is nearly 100 % in the course of electrolysis, and the voltage fluctuation at the completion of electrolysis is conspicuous.

⁽⁷⁾ The limit of measurement with each pour of the electrolytic solution is 20 μm to 25 μm with the chromium coating generally excellent in corrosion resistance. The electrolysis should preferably be stopped for every thickness of 25 μm for each coating film according to the accuracy of measurement, and the electrolytic solution should be renewed.

10.5 Factors influencing the measuring accuracy Following factors influence the measuring accuracy, therefore care shall be taken:

- a) coating thickness;
- b) electrolytic solution;
- c) composition of alloys.

Table 3 Application of electrolytic solution

Coating	Basis material (or substrate)				
	Iron	Copper and Copper alloys	Nickel	Aluminum	Zinc
Cadmium	No.1	No.1	—	—	—
Chromium	No.2	No.3	No.2 or No.4	No.2 or No.4	—
Copper	No.5	—	No.6	No.5 or No.6	No.7
Lead	No.8	No.8	No.8	—	—
Nickel	No.9	No.10	—	No.9	—
Silver	—	No.11	No.11	—	—
Tin	No.12	No.12	No.12	No.13	—
Zinc	No.14	No.14	—	—	—

Remarks: The numerical figures in the above table show No. of electrolytic solution in Table 4.

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Table 4 Examples of electrolytic solutions

No.	Composition of electrolytic solution	Informative reference
1	Dissolve 30 g of potassium chloride and 30 g of ammonium chloride in pure water to make 1 000 ml.	—
2	Dilute 95 ml of phosphoric acid (specific gravity 1.75) to 1 000 ml with pure water and add 25 g of chromic acid anhydride to it.	Used for a coating not less than 5 μm in thickness. Current density of about 100 mA/cm ² is preferable The error at that time is within $\pm 10\%$.
3	Dissolve 100 g of sodium carbonate in pure water to make 1 000 ml.	Used for a coating not less than 5 μm in thickness. Current density of about 100 mA/cm ² is preferable.
4	Dilute 64 ml of phosphoric acid (specific gravity 1.75) with pure water to make 1 000 ml.	Suitable for a thin decorative chrome coating. Current density of about 100 mA/cm ² is preferable.
5	Dissolve 800 g of ammonium nitrate in pure water and add 10 ml of ammonium hydroxide (specific gravity 0.88) to make 1 000 ml.	The thickness shows a value 1 % to 2 % lower than the true value.
6	Dissolve 100 g of potassium sulfate in pure water to make 1 000 ml and add 20 ml of phosphoric acid (specific gravity 1.75) to it.	—
7	Use fluosilicic acid solution with a content of not less than 30 vol%.	This solution joined by magnesium silicon fluoride is faintly acidic.
8	Dissolve 200 g of sodium acetate and 200 g of ammonium acetate in pure water to make 1 000 ml.	The thickness shows a value within 5 % higher than the true value.
9	Dissolve 800 g of ammonium nitrate in pure water to make 1 000 ml and add 50 ml of thiourea of 76 g/l to it.	The solution having elapsed 5 days shall not be used because of change in quality.
10	Dilute 100 ml of hydrochloric acid (specific gravity 1.18) with pure water to make 1 000 ml.	Current density is preferably about 400 mA/cm ² .
11	Dissolve 100 g of potassium fluoride in pure water to make 1 000 ml.	Not applicable to the plating including antimony or bismuth.
12	Dilute 170 ml of hydrochloric acid (specific gravity 1.18) with pure water to make 1 000 ml.	—
13	Pour 50 ml of sulfuric acid (specific gravity 1.84) into pure water cautiously to make 1 000 ml. Add 5 g of potassium fluoride to this solution to dissolve.	—
14	Dissolve 100 g of potassium chloride in pure water to make 1 000 ml.	—

11 Eddy current method for thickness test

11.1 Summary Pass a high frequency eddy current through a probe (measuring terminal), generate eddy current in the part of surface layer of the coating to be measured, measure the quantity of the eddy current to be changed by conductivity, thickness, shape and others, and thereby determine the thickness of the coating. Annex 1 (informative) shows the precautions in measurement by the eddy current method for thickness test.

11.2 Apparatus An example of apparatus is shown in Fig. 4.

11.3 Calibration The apparatus shall be sufficiently calibrated before use on a test. The following items shall be noted for precautions.

- a) The apparatus shall be calibrated according to the characteristics of apparatus by using a standard sample before use of this apparatus. Calibrations shall also be carried out during the measurement at suitable intervals of time.
- b) The standard sample for calibration shall be uniform in thickness and be covered with foil or a coating of a known thickness. As for foils, tight adhesion with substrate shall be cautiously verified.
- c) For calibration, the qualities of basis material and coating used as a standard of calibration shall be the same or similar to those of sample to be measured respectively.

11.4 Operation The operation shall be as follows.

- a) The apparatus shall be operated as directed in the instructions of each apparatus.
- b) Examine whether the combination of basis material and coating is measurable or not. Where no sufficient difference in conductivity is present between the two, the accuracy of measured value is extremely inferior or the measurement becomes impracticable.
- c) Select a suitable probe according to the thickness of basis material and the thickness of coating.
- d) Carry out the preparatory adjustment and calibration for each selected probe.
- e) Carry out measurements not less than three times at the same place in general and average three values excluding suspected values.
- f) Keep the pressure applied on the probe constant and make the probe contact vertically with the surface to be measured.
- g) Preferably avoid measuring at the end part or curved part of sample because of errors anticipated.

11.5 Factors affecting measuring accuracy The following factors shall be noted as affecting the measuring accuracy.

- a) Thickness of coating
- b) Magnetic property of basis metal

- c) Thickness and shape of basis metal
- d) Roughness of surface
- e) Edge effect

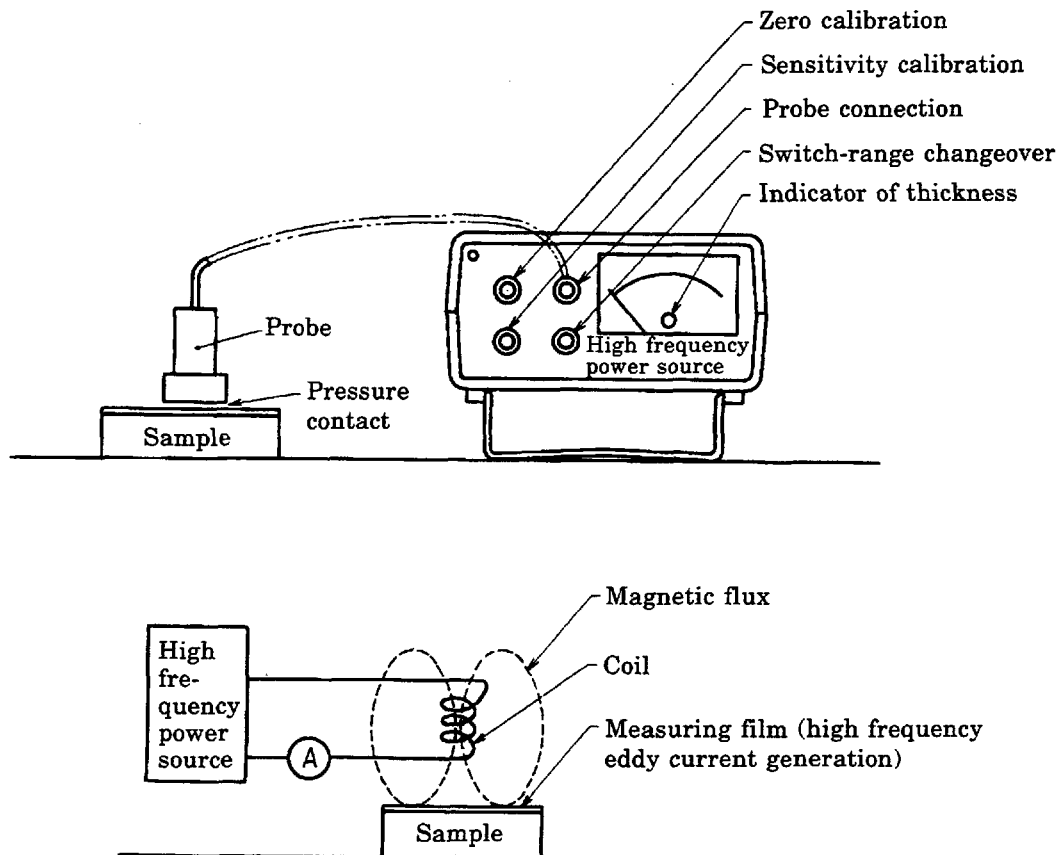


Fig. 4 Eddy current thickness measuring apparatus and its fundamental figure

12 Magnetic method for thickness test

12.1 Summary This is a method to obtain the thickness either by measuring the magnetic attraction between the magnet varying depending on the thickness of non-magnetic coating on the magnetic basis metal, or by measuring the magnetic reluctance of the magnetic flux passing through coating and magnetic basis metal. Precautions in measurement of the magnetic method for thickness test shall be indicated in Annex 2 (informative).

12.2 Apparatus Fig. 5 shows an example of the apparatus.

12.3 Calibration At the time of its use, sufficient calibration shall be made in accordance with 11.3. However, this step shall not be applied to the basis metals already magnetized.

12.4 Operation The operation shall be as follows.

- a) The operation shall be carried out as indicated in the handling instructions of each apparatus.

- b) The measurement shall be carried out at the same place where the calibration has been conducted and the probe shall also be applied with a definite pressure and in a definite direction.
- c) Carry out the measurement not less than three times usually at the same place and average three values except suspected values.

12.5 Factors affecting measurement accuracy The following factors shall be noted as affecting the measuring accuracy:

- a) Thickness of coating
- b) Magnetic property of basis metal
- c) Thickness and shape of basis metal
- d) Roughness of surface
- e) Edge effect

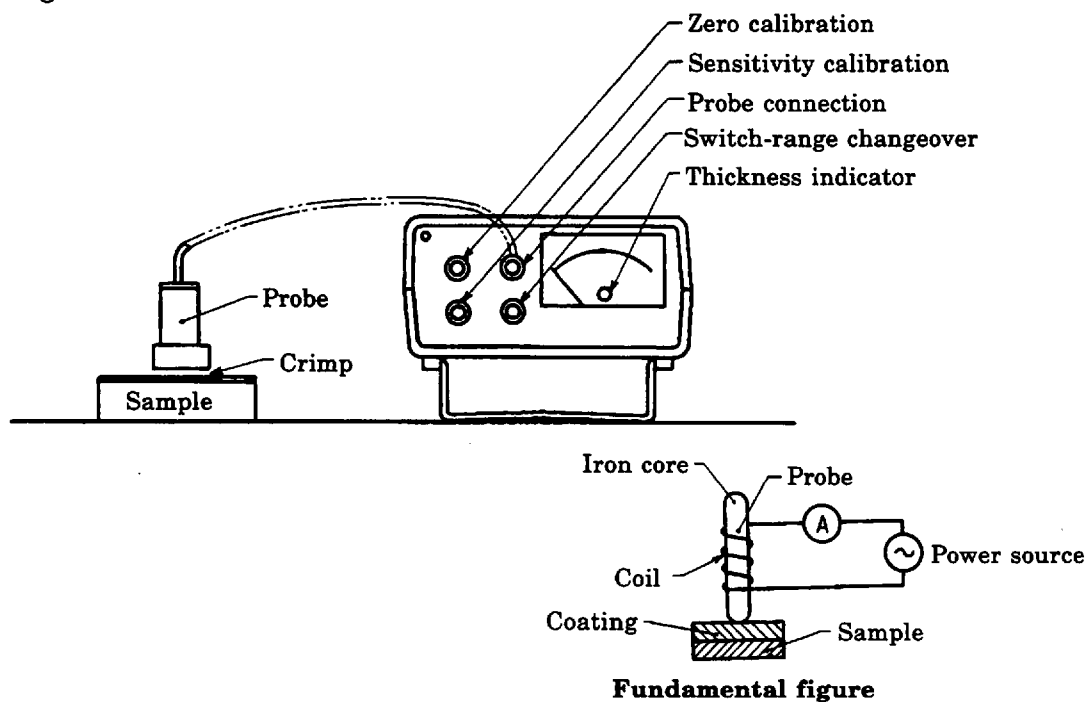


Fig. 5 Example of magnetic method for measuring thickness

13 X-ray spectrometric method for thickness test

13.1 Summary This is a method to obtain the thickness of coating by irradiating the sample with X-ray by means of X-ray spectrometric apparatus for measuring thickness by measuring the quantity of X-ray fluorescence radiated by the coating. Precautions in measurement by the X-ray spectrometric method for thickness test are indicated in Annex 3 (informative).

Remarks: The thickness of coating on basis metals or nonmetallic basis materials can be measured nondestructively.

13.2 Apparatus There are two types of the X-ray spectrometric apparatuses for measuring thickness: a wavelength dispersion type and an energy dispersion type.

An example of constitution of this apparatus is shown in Figs. 6 a) and b), respectively. In the case of energy dispersion type, the combination of each measurable basis material and coating partly varies depending on the provided detector, either a proportional counter tube detector or a semiconductor detector. Representative examples of combinations of basis material and coating which can be measured depending on types are shown in Table 5.

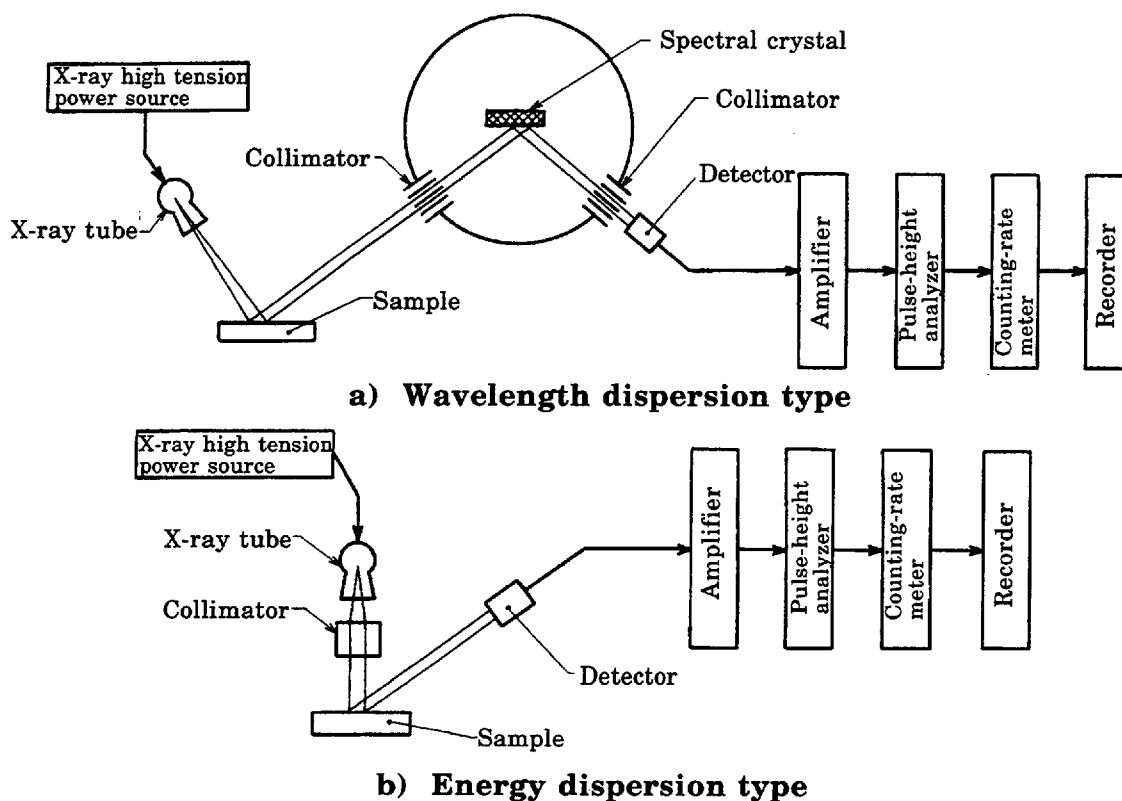


Fig. 6 Example of X-ray spectrometric apparatus for measuring thickness

13.3 Calibration At the time of use of the apparatus, sufficient calibration shall be made in accordance with 11.3.

13.4 Operation The operation shall be as follows.

- a) The apparatus shall be operated as indicated in the handling instructions of each apparatus.
- b) Verify whether or not the combination of basis material and coating is suitable for the measurement referring to Table 5.
- c) The thickness of coating shall not exceed the saturation thickness⁽⁸⁾ determined for each plating.
- d) The measuring time shall be determined by the quantity of X-ray fluorescence and the accuracy required.
- e) Measure at least three times at the same place of the sample in general and average three values except suspected values.
- f) Measurements at the end part and the curved part of sample shall be avoided as far as possible because of measuring errors anticipated.

Note (8) The “saturation thickness” means the thickness of film corresponding to 90 % amount of X-ray fluorescence when the coating is assumed to have an infinite thickness.

13.5 Factors affecting measuring accuracy The following factors shall be noted as affecting the measuring accuracy:

- a) Thickness of coating
- b) Quality and thickness of basis material and thickness of substrate coating
- c) Roughness of surface
- d) Curvature of sample

13.6 Safety management In operating the X-ray spectrometric apparatus for measuring thickness, safety management shall always be maintained not to leak X-rays.

Table 5 Representative of examples for measurable combinations of basis materials and coatings

Basis Material	Coating									
	Chromium	Nickel	Copper	Zinc	Rhodium	Silver	Cadmium	Tin	Gold	Tin-lead alloys
Aluminum	A	A	A	A	A	A	A	A	A	A
Iron	A	A	A	A	A	A	A	A	A	A
Nickel	A	C	B	B	A	A	A	A	A	A
Copper	A	B	C	B	A	A	A	A	A	A
Zinc	A	B	B	C	A	A	A	A	A	A
Silver	A	A	A	A	A	C	B	A	A	A
Brass	A	B	C	C	A	A	A	A	A	A
Nickel silver	A	C	C	C	A	A	A	A	A	A
Stainless steel	C	C	B	B	A	A	A	A	A	A
Plastic	A	A	A	A	A	A	A	A	A	A

Remarks: For the wavelength dispersion type and the energy dispersion type, “A” indicates “measurable” for both types and “C” indicates “unmeasurable” for both types. “B” indicates “measurable” for the wavelength dispersion type and also “measurable” for the energy dispersion type where the apparatus is equipped with a semiconductor detector, but “unmeasurable” where it is equipped with a proportional counter tube detector.

14 β -ray back-scatter method for thickness test

14.1 Summary This is a method to obtain the thickness of coating by measuring the quantity of back-scattered β -ray when the sample is irradiated with β -rays. The precautions in measurement by the β -ray back-scatter method for thickness test are indicated in Annex 4 (informative).

Remarks: The thickness of coating on the metallic basis material and the nonmetallic basis material can be measured nondestructively by this method. In view of the principle of measurement, in this method, the atomic numbers of basis material and the coating shall be sufficiently different by suitable numbers (usually not less than 3 or 4).

14.2 Apparatus An example of the composition of the apparatus is shown in Fig. 7 and the representative examples of combinations of basis materials and coatings which can be measured using this apparatus are shown in Table 6.

14.3 Calibration At the time of use of the apparatus, sufficient calibration shall be carried out in accordance with 11.3.

14.4 Operation The operation shall be as follows.

- a) The apparatus shall be operated as indicated in the handling instructions of each apparatus.
- b) Verify whether or not the combination of basis material and coating is suitable for the measurement referring to Table 6.
- c) Select a suitable β -ray source from Table 7 according to the type and thickness of coating. The higher the voltage of β -ray source is, the thicker coating can be measured.
- d) Confirm that the thickness of basis material is not smaller than the saturation thickness⁽⁹⁾.
- e) Confirm that the thickness of coating has not reached the saturation thickness in respect to the selected β -ray source.
- f) The measuring time shall be determined by the quantity of back-scattered rays from a sample and a required accuracy.
- g) Measure at least three times at the same place in general and average three values after excluding suspected values.
- h) The measurements at the end part and the curved part of sample shall be avoided as far as possible because of measuring errors anticipated. Where the curved part of sample is measured inevitably, care shall be taken to use the measuring jig suitable for that curvature.
- i) During the measurement, the sample and measuring jig shall be closely contacted.

Note (9) The minimum thickness which produces a phenomenon in which, on the basis of the incident rays from the β -ray source, only the absorbing rays and the back-scattered rays remain and no more change in the quantity of back-scattered rays is noticed.

14.5 Factors affecting measuring accuracy The following factors shall be noted as affecting the measuring accuracy.

- a) Thickness of coating and type of plating
- b) Quality and thickness of basis material, and thickness of substrate coating
- c) Roughness of surface

- d) Curvature of sample
e) Degree of adhesion of sample and jig

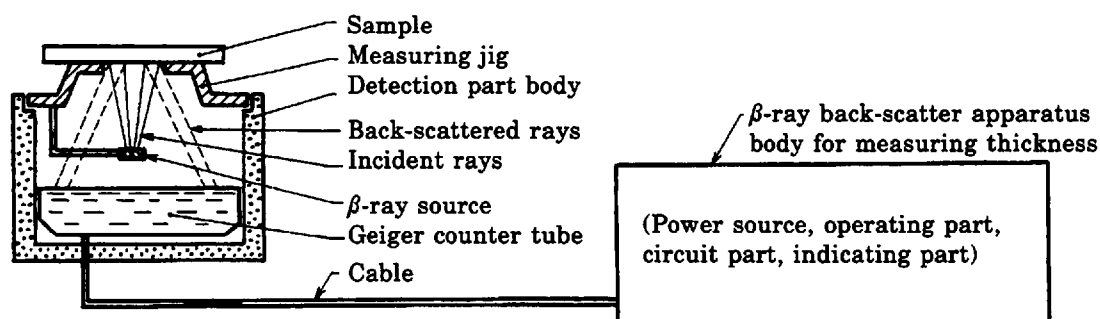


Fig. 7 Example of β -ray back-scatter apparatus for measuring thickness

Table 6 Representative examples of measurable combination of basis materials and coatings

Basis material	Coating							
	Nickel	Copper	Rhodium	Silver	Cadmium	Tin	Gold	Tin-lead alloys
Aluminum	○	○	○	○	○	○	○	○
Iron	-	-	○	○	○	○	○	○
Nickel	-	-	○	○	○	○	○	○
Copper	-	-	○	○	○	○	○	○
Zinc	-	-	○	○	○	○	○	○
Silver	○	○	-	-	-	-	○	○
Brass	-	-	○	○	○	○	○	○
Nickel silver	-	-	○	○	○	○	○	○
Stainless steel	-	-	○	○	○	○	○	○
Plastic	○	○	○	○	○	○	○	○

Remarks: The mark "○" in the above Table indicates "measurable" and the mark "-" indicates "unmeasurable".

At the time of use of the apparatus, sufficient calibration shall be carried out in accordance with 6.2.

Table 7 Examples of β -ray sources

Names of β -ray source	Voltage MeV	Half-life Year
Promethium (Pm-147)	0.22	2.6
Thallium (Tl-204)	0.77	3.5
Radium (RaD-E)	1.17	22.0
Strontium (Sr-90)	2.18	19.9
Ruthenium (Ru-106)	3.53	1.0

15 Multiple-beam interference test method

15.1 Summary This is a method to obtain the thickness of the coating from the deviation of the interference fringes caused by irradiating the monochromatic light beam on the sample and the reference plate, and reflecting the beam thereby using the multiple interference type thickness measuring device after dissolving and peeling a part of the coating of the sample, or forming a difference of levels by masking a part of the sample before coating. Precautions in the measurement of the multiple-beam interference test method are indicated in Annex 5 (informative).

15.2 Apparatus The apparatus to be used for this method shall be the Fizeau's multiple-beam interferometer type thickness measuring device.

15.3 Calibration The calibration is necessary for the multiple-beam interferometer method.

15.4 Measuring procedure The measuring procedure shall be as follows.

- a) **Preparation of difference of levels** Either as in the following 1) or 2).
 - 1) **Before coating** Protect a part of the sample from the coating in advance, and coat the sample, and peel the masked film. (masking)
 - 2) **After coating** Take the sample, clean the coated surface, and peel a part of the coating so as not to damage the basis material or the substrate.
- b) **Operation** The operation shall be carried out according to the handling instructions for each apparatus.

15.5 Factors affecting measuring accuracy The following factors shall be noted as affecting the measuring accuracy.

- a) Reflection by the coating
- b) Preparation and shape of the difference of levels
- c) Flatness of the sample
- d) Roughness of surface
- e) Cleanliness of the sample

16 Scanning electron microscope test method

16.1 Summary This is a method to obtain the thickness of the coating by observing the vertical section of the coating using the scanning electron microscope. Precautions in measurement by the scanning electron microscope test method are indicated in Annex 6 (informative).

16.2 Apparatus The apparatus to be used for this method shall be the scanning electron microscope specified in JIS K 0132. Fig. 8 shows an example of the apparatus.

16.3 Calibration At the time of use of the apparatus, sufficient calibration shall be carried out. Calibrations at appropriate intervals are necessary.

16.4 Operation The operation shall be carried out in accordance with the handling instructions for each apparatus.

16.5 Factors affecting measuring accuracy The following factors shall be noted as affecting the measuring accuracy.

- a) Roughness of surface
- b) Sample and inclination of its cross-section
- c) Contrast
- d) Magnification
- e) Stability of microphotograph

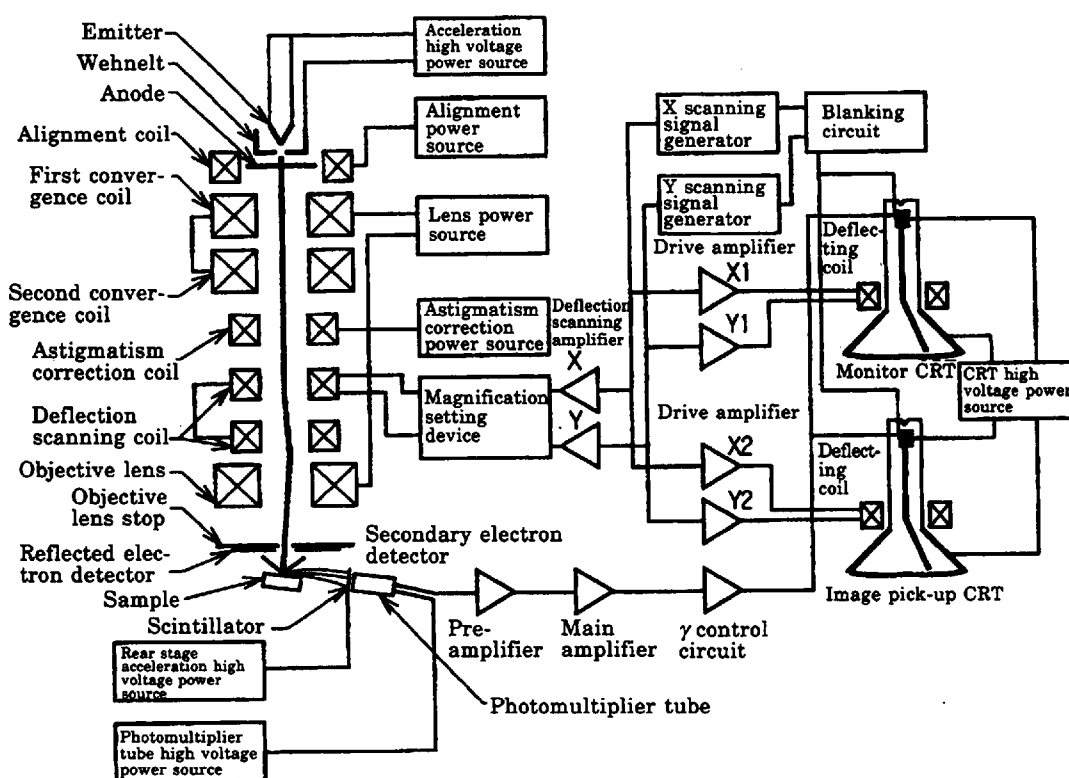


Fig. 8 Constitution of scanning electron microscope test apparatus

17 Micrometric method for thickness test

17.1 Classification of methods These are the micrometric methods for measuring the thickness of coating on the metallic or nonmetallic basis material.

- a) Coating destructive method
- b) Basis material destructive method
- c) Nondestructive method
- d) Profilometric method

17.2 Coating destructive method