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Replacing GB/T 6394-2002

Determination of estimating the average grain size of metal

金属平均晶粒度测定方法

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard replaces GB/T 6394-2002 "Determination of estimating the average grain size of metal".

As compared with GB/T 6394-2002, the main technical changes of this standard are as follows:

- ADD the terms and definitions of grain boundary, grain boundary intersection count, and intercept length (SEE Chapter 3);
- ADD the heat treatment method, sampling location, sampling orientation, polishing area requirements, and sample preparation requirements for the samples forming the austenite grains (SEE Chapter 6, Chapter 7);
- In the comparison method, ADD the requirements for the inspection parameters, view field selection, and rating operations details (SEE 8.1);
- ADD the requirements that the cementite outling grain display may use the pattern IV (SEE Table 2 in 8.1.1.3);
- MODIFY the values of the grain diameter and magnification corresponding to 400-timess and 500-timess rating chart; ADD the conversion values of 500-timess and 1000-timess the rating chart (SEE Table 3 and Table 4 of 8.1.2.3);
- In the area method, ADD the calculation formula of the rectangular measurement network; ADD the methods of field selection and counting precision (SEE 8.2.2);
- MODIFY the contents of the intersection method; ADD the various calculation formulas of the intersection method (SEE 8.3.1.8);
- ADD the calculation method of the non-equiaxed grains; DEFINE the calculation process (SEE Chapter 9);
- As for the assessment of the two-phase and multi-phase grains, ADD the requirements for the determination report of the phase characteristics and proportion; ADD the intercept method (SEE 10.4.3);
- ADD the report content requirements for the basic data, non-equiaxed grains, and multi-phase grain through the area method and the intersection method; IMPROVE the grain size report (SEE Chapter 11);
- ADD the precision and deviation requirements (SEE Chapter 12);

Determination of estimating the average grain size of metal

1 Scope

1.1 This standard specifies the expression and determination methods of the average grain size of metal structure, including the comparison method, area method and interception method; it is applicable to the single-phase structure; however, after being specified, it is also applicable to the determination of the average grain size of the certain type in the multi-phase or multi-component sample. If the histomorphology of the non-metallic material is similar to the metal structure in the rating chart, it may also make reference to it.

1.2 This standard uses the grain area, diameter or intercept length of the unimodal distribution (similar to the lognormal distribution) to determine the average grain size of the sample, AND it is not applicable to the grain size of the bimodal distribution. The evaluation of double grain size is as shown in GB/T 24177. For the determination of individual coarse grains on a fain grain substrate, SEE YB/T 4290.

1.3 This standard applies only to the measurement of the plane grain size measurement, AND it is not applicable to the three-dimensional grain size, that is, the measurement of three-dimensional grain size.

1.4 This standard is only used as a recommended test method, AND it shall cannot determine whether the test material is received or suitable for the range of use.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this Standard.

GB/T 4335 Determination of ferrite grain size for cold rolled low carbon steel sheets

GB/T 13298 Inspection methods of microstructure for metals

GB/T 24177 Standard test methods for characterizing duplex grain sizes

GB/T 30067 Standard terminology relating to metallography

YB/T 4290 Test methods for estimating the largest grain observed in a metallographic section (ALA grain size)

3 Terms and definitions

The terms and definitions as defined in GB/T 30067 AND the following terms and definitions apply to this document.

3.1

Grain boundary

It refers to, for the polycrystalline material, a narrow region transitioning from one crystal direction to the other crystal direction, thereby separating the adjacent grains.

3.2

Grain

It refers to the entire area surrounded by grain boundaries, that is, the area within the original grain boundary observed on the two-dimensional plane, or the volume surrounded by the original grain boundery on the three-dimensional object. For twin-grain boundary materials, the twin-grain boundary is not considered.

3.3

Grain size

It refers to the measurement of the size of grain, which is evaluated or determined by different methods, is usually expressed using the length, area, volume, or grain size level, AND the grain size as expressed using the grain size level is independent of the measurement method and the unit of use.

3.4

Micro-grain size number

G

The micro-grain size number G is defined as:

$$N_{100} = 2^{G-1} \qquad \cdots\cdots (1)$$

3.5

Macro-grain size number

Through reasonable count, it may reach to the precision of ± 0.25 level. The results of the area method are unbiased, AND its reproducibility and repeatability are less than ± 0.5 level. The key to the precision of the area method is that during counting, it must indicate the counted grain.

5.4 Intersection method: intersection method is to count the number of the intercept between the test line (or grid) of known length and the grain or the number of intersection between the test line (or grid) and the grain, AND to calculate the unit length intercept count N_L or the intersection count P_L, so as to determine the grain size level G. The precision of the intersection method is a function of the intersection or intercept count, which can achieve a precision of better than ± 0.25 level by an effective count. The results of the intersection method are unbiased, AND the reproducibility and repeatability are less than ± 0.5 level. For the same level of precision, since the intersection method can count accurately without the need of marking, it is faster than the area method.

5.5 For equiaxed grain samples, it is convenient and practical to use the comparison method to evaluate the grain size. For the inspection of mass production, its precision is sufficient. For the determination of the average grain size requiring for higher precision, it may use the area method and the intersection method. The intersection method is more effective for the sample of elongated grain composition.

5.6 In the event of a dispute, the intersection method is used as the arbitration method.

5.7 Low-carbon steel cold-rolled sheet ferrite grain size is determined in accordance with the provisions of GB/T 4335.

5.8 A single grain cannot be determined on the basis of the standard rating chart. Because the composition of the standard rating chart takes into account the relationship between the cut plane and the grain three-dimensional arrangement, it shows the arrangement distribution of grain from the minimum to the maximum, which reflects the representative normal distribution results. Therefore, the rating chart cannot be used to determine a single grain.

5.9 When determining the grain size, it shall be recognized that the determination of grain size is not a very accurate measurement. Because the metal structure is the accumulation of three-dimensional grain of different sizes and shapes. Even if the size and shape of these grains are the same, the size of the grains distributed through any section (test surface) of the structure will vary from the maximum to the zero. Therefore, there can be neither grain distribution of absolute uniform size on the test surface, nor two identical test surfaces.

5.10 The grain size and position in the microstructures are randomly distributed. The randomly measured average grain size is only representative if moving the field of view without intention and placing the measurement grid. If moving the field of view at a certain part of a sample, the concentrated measurement will produce poor representativeness. The so-called

8.1.1.4 The correct judgment of the observer requires selecting the magnification factor used, the appropriate test surface size (grain number), the number and position of the sample's representative section, the determination characteristics, AND the field of view used for the average grain size. It shall not visually select an evaluated area that seems to be an average grain size. It is recommended to follow the requirements of 5.10 to select the field of view.

8.1.1.5 As for the evaluation of grain size, it shall randomly select three or more representative fields of view from the sample section to measure the average grain size, AND report it using the level which best represents the grain size distribution of the sample.

8.1.1.6 If the grain is found to be uneven in the sample, after overall observation, if it is an accidental or individual phenomenon, it may not be calculated. If it is common, it shall calculate the area percentage of different levels of grain in the field of view. If the area occupied by the dominant grain is not less than 90% of the area of the view field, it shall only record the number of levels of this grain. Otherwise, it shall use different level numbers to represent the grain size of this sample, wherein the first level number represents the level of the dominant grain. In case of double grain size, it shall be evaluated in accordance with GB/T 24177; in case of individual coarse grain, it may be evaluated in accordance with YB/T4290.

8.1.1.7 When using the comparison method, if it requires re-inspection, it may change the magnification factor, in order to overcome the subjective bias that may be included in the initial inspection results.

8.1.2 Evaluation of micro-grain size

8.1.2.1 It generally use the magnification factor same as that of the corresponding standard series rating chart, for direct comparison. USE the grain structure image or micrograph of the representative field of view to compare with the corresponding series rating charts of Table 2 or the duplicated transparent film of the standard rating chart, SELECT the standard rating chart number or grain size which is most close to the test image; RECORD the evaluation results. Between the two standard images of two integer levels, RECORD the average of two picture levels.

8.1.2.2 If the tested grain image and the standard series rating chart are projected onto the same screen, it can improve the rating precision.

8.1.2.3 When the measured grain size exceeds the range included in the standard series rating chart, OR otherwise the reference magnification factor (75-timess or 100-timess) cannot meet the requirements, it may use other magnification factors, which is converted through Table 3 and Table 4 OR using the formula (3) as given in 8.1.2.4. Typically, the magnification factor selected is a simple integer multiple of the reference magnification factor.

8.1.2.4 If the other magnification factor M is used for comparative evaluation, COMPARE the tested grain image of the magnification factor M with the series

From the polishing surface, randomly SELECT the field of view from different locations, to ensure trueness and validity. To ensure that the average value is valid, it shall calculate at least 3 fields of view.

8.3 Intersection method

8.3.1 General requirements

8.3.1.1 The intersection method is to determine the grain size by counting the number of intercepts between the measuring line (or grid) of given length and the grain boundary.

8.3.1.2 The intersection method is simpler than the area method. It is recommended to use the manual counter, to avoid routine errors during counting AND to eliminate deviations that may occur when the count is higher or lower than expected.

8.3.1.3 It shall use the intersection method for the non-uniform equiaxed grains. For non-equiaxed grain size, the intersection method can be used not only to respectively determine the grain size of three mutually perpendicular directions, but also to calculate the overall average grain size.

8.3.1.4 The intersection method is divided into straight-line intersection method and circular intersection method. The circular intersection method can automatically compensate the error produced by the deviation from equiaxed grain without the needs of adding too many field of view, so it overcomes the issues that the intersection at the test line end is not apparent. The circular intersection method is more appropriate as a quality testing method to evaluate the grain size.

8.3.1.5 It is recommended to use the 500 mm measuring grid, AND its size is as shown in Figure 1.

of view. The selection of the field of view shall be as large as possible on the test surface of the sample.

8.3.2.4 For obvious non-equiaxed grains, such as the moderately processed materials, the dimensions are measured separately by the parallel beams along the 3 principal axis of the sample, to obtain more data. Usually the longitudinal direction and transverse direction are used, AND the normal direction is also used if necessary. Any 100 mm line segment in Figure 1 can be moved in a parallel manner to the position marked with "+" in the same image for 5 times for use.

8.3.3 Single circle intersection method

8.3.3.1 For materials with significant differences in grain size at different locations on the sample, it shall use the single circle intersection method, in which case a large number of field of view measurements are required.

8.3.3.2 The grid measuring circle used may be of any circumference, usually it is 100 mm, 200 mm and 250 mm, AND it may also use the circle identified in Figure 1.

8.3.3.3 SELECT the appropriate magnification to ensure that each circumference produces about 35 intersections. When the measuring grid passes through the confluence point of three grains, it is counted as 2 intersections.

8.3.3.4 DISTRIBUTE randomly the required number of circumferences on the test surface as large as possible; INCREASE the number of field of view until sufficient precision is obtained.

8.3.4 Three circle intersection method

8.3.4.1 The tests show that reliable precision can be obtained when the intersection count of each sample reaches 500. The measurement data is subjected to X^2 test, AND the result shows that the intersection count is subjected to a normal distribution, so it is allowed for the measurement value to be subjected to the statistic processing of normal distribution. For each grain size determination result, it may calculate the result deviation and the confidence level (SEE Appendix B).

8.3.4.2 The measuring grid consists of three circles of concentric equidistant and total circumference of 500 mm, as shown in Figure 1. USE this grid to measure any of the five different fields of view which are randomly selected, and RECORD the intersection count for each. And then CALCULATE the percentage of count relative error, the average grain size, and the confidence interval. In general, the relative error percentage of 10% or less is an acceptable level of precision. If the relative error percentage cannot comply with the requirements, it is required to increase the number of fields of view, until the relative error percentage complies with the requirements.

For most industrial production tests, if the second phase is substantially the same as the substrate grain size, which is composed of islands or blocks; OR otherwise the number of grain of the second phase is smaller and the size is very small, which are mainly located at the primary grain boundary, it may use the comparison method.

10.3 Area method

If the substrate grain boundaries are clearly visible AND the second phase particles are predominantly between the substrate grains rather than within the grains, it may use the area method to measure the percentage of the second phase in the inspection area. In general, firstly MEASURE the total number of the second phase which is of the minimum content, then USE the difference to measure the substrate phase; FOLLOW 8.2 to calculate the number of substrate grain which is totally falling into the inspection area as well as the intersection counts between the substrate grain and the inspection area boundary. The area of the selected measuring grid shall be such as able to cover the substrate grain only. USE the grain number within the substrate unit net area to determine the effective average grain size. FOLLOW the method of Appendix B, in the measured value of each field of view, to statistically analyze the grain number $N_{A\alpha}$ per unit area of the substrate (α). Then USE the Table 6 or the formula (9) to determine the effective grain size of the substrate from the total average value $\overline{N}_{A\alpha}$.

10.4 Intersection method

10.4.1 The restrictive provisions in the 10.3 area method also apply to this intersection method. In addition, it shall also follow 10.3 to determine the percentage of the substrate phase. USE one or more test circles in Figure 1 to form the measuring grid, to count the number of the substrate grain N_α which is intersected with the test line. USE the formula (23) to determine the average intercept length of the substrate phase.

$$\overline{l}_\alpha = \frac{(V_{V\alpha})(L/M)}{N_\alpha} \quad \cdots\cdots(23)$$

Note: It may use $A_{A\alpha}$ to estimate $V_{V\alpha}$.

10.4.2 USE the Table 6 or the formula (15) to determine the substrate grain size (α). In fact, it is difficult to determine the area fraction of the substrate (α) and the intersection count of the substrate (α) and the test line by manual operation for each field of view. It may determine the average intercept length of each view field substrate (α), AND perform statistical analysis against the data of each view field in accordance with the methods of Appendix B. If the $V_{V\alpha}$ and N_α cannot be measured at the same time for the same field of view, then it can only perform statistical analysis of the $V_{V\alpha}$ and N_α data.

10.4.3 Randomly PLACE a straight parallel test line on the grain image, AND it may calculate the average intercept length $\overline{l}_\alpha$ by measuring each intercept.

11.1.7 For double grain size, FOLLOW the requirements of GB/T 24177 to report two typical representative grain sizes; and for ALA grain size, it is evaluated in accordance with YB/T4290.

11.2 Content of report

11.2.1 As for the grain size of ferritic and austenitic steels, the test report shall include:

a) Standard number;

b) Steel designation and furnace numbers;

c) Product specifications and dimensions;

d) Teat treatment methods of the sample (formation method of the original austenite grains of ferrite steel);

e) Grain size display method;

f) Grain size evaluation method;

g) Grain size value (SEE 11.1.3 ~ 11.1.7 to report the relevant content);

h) Test report number and date;

i) Name of the examiner.

11.2.2 As for the grain size of copper and copper alloy, it is preferable for the test report to include the following:

a) Grain size display method;

b) Grain size is reported in millimeters by average diameter.

Note: The average diameter refers to the average “diameter” of the grains displayed on the test surface.

11.2.3 Grain size display method and grain size of other metals and their alloys are reported in accordance with the relevant technical conditions or the requirements of both parties.

12 Precision and deviation

12.1 Grain size measurement precision and deviation depends on the representation of the sample AND the polished surface selected for measurement. If the grain size changes in the product, the selection of the sample and the field of view shall fully reflect such change.

12.2 The relative precision of the product grain size measurement increases as the number of samples taken from the product is increased. The relative

Appendix A

(Normative)

Grain size formation and display method

A.1 Austenite grain size of ferritic steels

A.1.1 Austenite grain formation of ferritic steels

A.1.1.1 Scope

When measuring the grain size, it is sometimes necessary to show some of the previous characteristics of the grains (for example, the test of austenite grains of high temperature status in the cold state), it is required to perform corresponding processing or technology operation against the material sample, in order to show the existed austenite grain boundaries in the material in the original austenitizing process. This Appendix is for recommendations.

A.1.1.2 Formulation method

Unless otherwise specified, the original austenite grain size shall be formed by one of the following methods. The carbon content indicators in the following methods are only recommended. There are many ways to form the original austenite grain size, AND understanding the knowledge on grain growth and coarsening is helpful to determine selecting which method. For any kinds of steel, the size of the original austenite grain is mainly determined by the steel heating temperature and the holding time at that temperature. It shall be noted that the heating atmosphere may affect the growth of the grains on the outer layer of the sample. The original austenite grain size will also be affected by the original steel treatment, such as austenitizing temperature, quenching, normalizing, hot working and cold processing. Therefore, in the determination of the original austenite grain size, it shall consider the effects of the original and subsequent treatment on the sample.

A.1.1.2.1 Correlation procedure

For carbon and alloy steels, the test conditions shall be related to the heat treatment system used to improve the performance when the material is actually used. As agreed between both parties, the conventional process is that the sample heating temperature does not exceed the normal heat treatment temperature of 30 °C, BUT the maximum does not exceed 930 °C, AND the holding time is 1 h ~ 1.5 h. The cooling rate is determined by the method of heat treatment AND it is microscopically tested in accordance with Table 2.

A.1.1.2.2 Carburizing procedure

b) The medium carbon steel (carbon content of about 0.50%) is suitable to be furnace cooled;

c) As for the carbon steel having a higher carbon content AND the alloy steel having a carbon content higher than 0.40%, it is recommended to heat the sample to 860 °C, maintain this temperature for 30 min, reduce the temperature to 730 °C ± 10 °C, maintain this temperature for 10 min, AND then water cooled or oil cooled.

A.1.1.2.5 Oxidation procedure

It is applicable to the carbon steel and alloy steel having a carbon content of 0.25% ~ 0.60%. POLISH one surface of the sample (it is recommended to use about 400 grain size or 15 μm abrasive). PLACE the sample in the furnace with the polished surface facing upwards. Unless otherwise specified, if the carbon content (mass fraction) is not more than 0.35%, the sample is heated at 890 °C ± 10 °C; if the carbon content (mass fraction) is more than 0.35%, the sample is heated at 860 °C ± 10 °C, maintained at this temperature for 1 h, and quenched in cold water or salt water. The polished and quenched sample shows the austenite grain size on the oxidized surface.

A.1.1.2.6 Direct hardening procedure

It is applicable to carbon steel and alloy steel having a carbon content usually less than 1.00%. Unless otherwise specified, if the carbon content (mass fraction) is not more than 0.35%, the sample is heated at 890 °C ± 10 °C; if the carbon content (mass fraction) is more than 0.35%, the sample is heated at 860 °C ± 10 °C, maintained at this temperature for 1 h, and quenched at the complete hardening speed.

After cooling, the sample surface is cut, ground, polished, and etched to show the martensite. Before etching, it may be heated at 230 °C ± 10 °C, maintained at this temperature for 15 min, and tempered, so as to improve the contrast.

A.1.1.2.7 Cementite outling procedure

It is applicable to the carbon steel and alloy steel having a carbon content more than 1.00%. Usually the sample having a diameter or side length of about 25.4 mm is used for the test. Unless otherwise specified, the sample is heated at 820 °C ± 10 °C, maintained at this temperature for 30 min, AND then cooled to below the lower critical temperature at a sufficiently slow cooling rate in the furnace, in order to precipitate the carbide from the austenite grain boundary. After cooling, the sample surface is cut, ground, polished and appropriately etched to show the original austenite grain size delineated by the carbides precipitated at the grain boundary.

A.1.1.2.8 Fine pearlite outling procedure

It is applicable to the eutectoid steel for which the steel is slightly lower than or slightly higher than the eutectoid composition. There are two ways:

grain morphology, to show the original austenite grain size. Generally, the nitric acid alcohol is used to etch the sample to show the ferrite outling AND evaluate the grain size. Commonly used etchants are:

a) 3% ~ 4% nitric acid alcohol solution;

b) 5% picric acid ethanol solution.

A.1.2.1.3 Grain displayed by grain boundary oxidation

The steel is heated in an oxidizing atmosphere AND the oxidation takes precedence along the grain boundary. Therefore, the commonly used method is to grind a metallographic polished surface, heat it in the air at the specified temperature for a required period, AND then slightly grind it to remove the oxide scale, so that the original austenite grain boundary is shown due to the presence of the oxide. For clarity, it may use 15% hydrochloric acid ethanol solution to etch it. In accordance with the oxidation, the sample is tilted appropriately by 10 ° ~ 15 ° for grinding and polishing, so that the austenite grains of the oxide layer are shown as complete as possible.

A.1.2.1.4 Martensite grain displayed by fine pearlite (troosite)

It is a method particularly applicable to eutectoid steel, which is difficult to identify grains if using other methods. In a fully hardened and unhardened transition zone, there is a small region that is not completely hardened. In this region, the original austenite grains are composed of martensite grains surrounded by a small amount of fine pearlite (troosite), thereby showing the original austenite grain size. This method can also be applied to some steel slightly below or slightly higher than the eutectoid composition. Commonly used etching solution are:

a) 3% ~ 4% nitric acid ethanol solution;

b) 5% picric acid ethanol solution.

A.1.2.1.5 Etching of martensite grains

There are two ways:

a) As for the completely hardened martensitic steels, the use of the etchant which can enhance the contrast of difference between martensite grains may show the grain size of the original austenite. Before etching, the tempering at 230 °C for 15 min may improve the contrast. It is recommended to use such reagents: 1 g of picric acid, 5 mL of HCl (specific gravity 1.19), and 95 mL of ethanol;

b) USE the etchant that preferentially displays the original austenite grain boundary. The saturated aqueous solution of picric acid containing corrosion inhibitor has a better effect. Recommended etchant: A saturated aqueous solution of picric acid containing tridecylbenzene sulfonate (or dodecyl) wetting agent.

A.3.2.1 Stable materials

At room temperature, the sample as an anode is electrolytically etched in a 60% nitric acid aqueous solution having a volume concentration of 60%. In order to reduce the occurrence of twin grain, it shall use the low voltage (1 V ~ 1½ V). It is also recommended to use this method to show the ferrite grain boundaries in ferritic stainless steels.

A.3.2.2 Unstable materials

The grain boundaries are displayed by precipitated carbides by heating at a temperature of 480 °C ~ 700 °C in the sensitized temperature range. The grain shape is shown using an etchant displaying the corresponding carbides. Commonly used etchants are:

a) The nitric acid aqueous solution of volume concentration 60%;

b) Copper sulfate aqueous solution: 5 g of copper sulfate ($CuSO_4 \cdot 5H_2O$) + 20 mL of hydrochloric acid + 20 mL of water;

c) 10% oxalic acid electrolysis: 10 g of oxalic acid + 100 mL of water; the voltage is 6 V, AND the time is 15 s ~ 60 s.

Note: In order to show the grain size of the austenitic material, it shall show the grain size by the appropriate etching method. It is important to recognize that the tendency of twin grain will confuse the grain size evaluation, and it is preferable for the amount of etching solution to apparently minimize the twin grain amount.

Appendix B

(Normative)

Statistical technique - Calculation of confidence interval and relative error of grain size measurement results

B.1 Overview

B.1.1 It is recommended to use the statistical analysis method of this Appendix for data processing of the intersection method, to ensure that the measurement results meet the requirements of the corresponding confidence interval and relative error.

B.1.2 Grain size measurement cannot be a very accurate measurement, so only the determination of which the precision is calculated is considered complete. Since the selected field of view for measurement is limited AND the sample number is small, this Appendix uses the t distribution statistical method to process the measured data. The use of 95% confidence interval (95% CI) means that the measurement results have a possibility of 95% falling into the indicated confidence interval. In this precision range, in accordance with the normal confidence degree, it may consider that the measured grain size represents the true average grain size of the sample under test. This standard uses normal confidence to express the expectation that 95% of the actual error falls within the specified uncertain error. In this example, it calculates the average grain size through determining the selected n "representative" fields of view, AND it shall not be considered as a fixed and reliable representative value of grain size. It shall use the statistical method for data processing, use the standard deviation to conduct a series of calculation, AND estimate the confidence interval (95% CL) in which the measured average grain size falls.

B.1.3 There is a significant change in the grain size between the different fields of view for many samples, AND such change is the major cause for uncertainty. Using the manual method which can obtain the required precision can prove that the precision of a single count is comparable to that of natural fluctuation. The use of the counter method can guarantee local high precision, unless determining many fields of view, otherwise it can only make small improvement for the entire precision, BUT it is helpful to distinguish between natural fluctuations and counting errors.

B.1.4 CALCULATE the relative error %RA, AND when %RA is not more than 10, the measurement result is considered valid.

B.2 Calculation method

At the magnification M, USE the measuring grid having a length of L (mm); based on the measurement results, CALCULATE the relative error (% RA) of

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