Evaluation of Uncertainty in Determining Average Grain Size by ASTM E112 Standard

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Abstract. In this paper, the ASTM grain size number of aluminium alloy specimen was determined respectively by Abrams Three-Circle Procedure and Planimetric Procedure of ASTM E112-13 Standard, and the uncertainty of the result was evaluated. The results are as follows: 1) Using the Abrams Three-Circle Procedure determines $G$, $G=4.13$, $U=0.26$, $k=2$; Using the Planimetric Procedure determines $G$, $G=3.94$, $U=0.15$, $k=2$. 2) It is suggested that the ASTM E112 standard should provide that the errors of counting grain boundary intersection count or number of grains should be less than or equal to 2, and the ruler that determines the diameter of test circle or scale length should be with the scale that is less than or equal to 0.10 mm. On this condition, the uncertainties caused by them in determining $G$ can be negligible. 3) The main sources of uncertainty for the determination of $G$ by Abrams Three-Circle Procedure and Planimetric Procedure are repetitive measurement, which depends on the uniformity of grain size of specimen. 4) The $\%RA$ limit of ASTM E112 for determining $G$ should be redefined according to the measurement uncertainty in order to be up to the precision requirement ($\leq 0.25$).

1. Introduction

Metallographic examination is a very important testing field in metal material testing. In the field of metallographic examination, the semiquantitative comparison method is the main detection method, and the quantitative analysis items are very few. The ASTM E112-13 Standard test methods for determining average grain size\textsuperscript{[1]} provides three methods for average grain size determination: comparison procedure, intercept procedures and planimetric procedure. The intercept procedures and planimetric procedure are very typical quantitative methods.

According to the ISO/IEC 17025-2017 Accreditation criteria for the competence of testing and calibration laboratories\textsuperscript{[2]}, Laboratories shall identify the contributions to measurement uncertainty. When evaluating measurement uncertainty, all contributions which are of significance, shall be taken into account using appropriate methods of analysis. A laboratory performing testing shall evaluate measurement uncertainty. Where the test method precludes rigorous evaluation of measurement uncertainty, an estimation shall be made based on an understanding of the theoretical principles or practical experience of the performance of the method.
In the field of chemical analysis and mechanical properties testing of metal materials, uncertainty evaluation of measurement results is widely used. In the two fields, there are many published papers about the uncertainty evaluation and standardization has been achieved \cite{3-4}. In the field of metallographic examination, the uncertainty evaluation of measurement results is seldom used. The main reasons are as follows: 1) the semiquantitative comparison methods are difficult to be rigorous evaluated; 2) The quantitative metallographic examination methods are few; 3) there are hardly any quantitative metallographic results in product standards as a method of evaluating product eligibility. However, through the evaluating measurement uncertainty, the main contributions to measurement uncertainty could be identified. From the management point of view, the testing quality can be effectively controlled. From the technical point of view, it can not only further perfect the standard test method, but also improve the accuracy of the standard test method.

In this paper, the ASTM grain size number of aluminium alloy specimen was determined respectively by Abrams Three-Circle Procedure and Planimetric Procedure of ASTM E112-13 Standard. The test circletotal circumference used in Abrams Three-Circle Procedure is 500 mm, and the test circle area used in Planimetric Procedure is 5000 mm$^2$. And the uncertainty is evaluated and expressed according to the JJF 1059.2-2012 Evaluation and Expression of Uncertainty in Measurement\cite{5}.

2. Testing scheme
The test specimen is aluminium alloy (AlSi1). The polished surface is 18 mm x 10 mm. The film on the surface of specimen is prepared by an oxidation. In the whole polished surface, the grain sizes are observed and the images are captured under polarized light randomly, and there are no overlapping fields. The magnification of objective lens is 10x. The selection of magnification ensures that the grain boundary intersection counts are 50 to 100 and the numbers of grains are not less than 50.

The quantitative detection methods of G are as follows: 1) Abrams Three-Circle Procedure, the designed diameters of three test circles are 79.58 mm, 53.05 mm, and 26.53 mm. 2) Planimetric Procedure, the designed diameter of test circle is 79.80 mm. In the captured metallographic photographs, the scale and test circle are superimposed, and then the 100 × photographs are printed. On the printed photographs, measuring the scale length and test circle diameter, counting the grain boundary intersection counts of three circles, and counting the numbers of grains completely within the test circle and the numbers of grains intercepted by the test circle.

Using the vernier calipers with 0.02 mm scale to determine the scale length or test circle diameter. The scale length is measured twice and the diameter of the circle is measured twice in the horizontal and vertical directions, and then the average scale length or average diameter of circle is calculated. When counting grain boundary intersection count or number of grains, appropriate labelling on the printed photographs is used to reduce the statistical counting error.

For the Abrams Three-Circle Procedure, the ASTM G is calculated from:

$$G = 6.643856 \lg \left( \frac{M}{L} \right)^P - 3.288 \quad (1)$$

Where $M$ is the magnification, $L$ is the test circle total circumference in mm and $P$ is the intersection counts.

For the Planimetric Procedure, the ASTM G is calculated from:

$$G = 3.321928 \lg \left( \frac{M}{A} \right)^N - 2.954 \quad (2)$$

Where $M$ is the magnification, $A$ is the test circle area in mm$^2$ and $N$ is the total number of grains.

3. Test result
Figure. 1a) is a metallographic photograph superimposed with the three circles and with a scale length of 200 μm for the Abrams Three-Circle Procedure. Figure. 1b) is a metallographic photograph superimposed with the circle and with a scale length of 200 μm for the Planimetric Procedure.
The average scale length on Fig.1 is 23.30 mm, and the range of the two measurements is 0.02 mm. The actual magnification of metallographic photograph is 101.50x, that is, M=101.50x in formulas (1) and formulas (2), and the magnification is within 2% of the permissible error of ASTM E112. The average diameters of the three circles on Fig. 1a are 79.48 mm, 52.96 mm, and 26.32 mm, respectively. The range of the two measurements for each circle is 0.02 mm. The actual total circumference of the three circles is 498.76 mm, that is, L = 498.76 mm in formula (1), and the relative error between actual total circumference and design total circumference is 0.25%. The actual magnification of metallographic photograph is 101.50x, that is, P = 101.50x in formulas (1) and formulas (2). The relative error between actual area and design area is 0.15%. The average magnification of metallographic photograph is 101.50x, that is, P = 101.50x in formulas (1) and formulas (2). The relative error between actual area and design area is 0.15%. The average circle diameter on Fig. 1b is 79.86 mm. The actual ci error between actual total circumference and design total circumference is 0.02 mm. The range of the two measurements for each circle is 0.02 mm. The actual total circumference of the three circles is 498.76 mm, that is, L = 498.76 mm in formula (1), and the relative error between actual total circumference and design total circumference is 0.25%.

Table 1 shows the original data of average grain size determined by Abrams Three-Circle Procedure. In Table 1, P_i is the total grain boundary intersection count, P_M is the medium circle grain boundary intersection count, P_S is the small circle grain boundary intersection count, P_i is the total grain boundary intersection count on a metallographic photograph, and P = P_i + P_M + P_S. P is cumulative grain boundary intersection count on several metallographic photographs, P_i is average grain boundary intersection count, and S_i is standard deviation of P_i. t_{95%}(v) is the quantile of t-distribution under the n fields (degree of freedom v = n-1) and confidence probability 95%. 95%CI is 95% confidence interval of several P_i. %RA is percent relative accuracy under 95% confidence probability, that is 95%CI/P. G is the AST grain size number obtained under %RA limit of several metallographic photographs. According to the requirements of ASTM E112, when %RA is less than or equal to 10%, the G result is acceptable. After the tenth field has been detected, \( \sum P_i = 653 \), P = 65.3, 95% RA = 4.13. For the aluminium alloy (AlSi1) specimen, the ASTM grain size number is 4.13 determined by Abrams Three-Circle Procedure.

Table 1. The Abrams Three-Circle Procedure results

| field | P_L | P_M | P_S | P_i | \( \sum P_i \) | P | S_i | t_{95%}(v) | 95%CI | %RA | G |
|-------|-----|-----|-----|-----|-------------|---|-----|-------------|-------|------|---|
| 1     | 37  | 28  | 14  | 79  | 79          | - | -   | -           | 4.68  |      |   |
| 2     | 33  | 23  | 13  | 69  | 148         | 74.0 | 7.07 | 12.706 | 63.53 | 85.85% | 4.49 |
| 3     | 24  | 15  | 10  | 49  | 197         | 65.7 | 15.28 | 4.303 | 37.95 | 57.79% | 4.14 |
| 4     | 29  | 21  | 11  | 61  | 258         | 64.5 | 12.69 | 3.182 | 20.19 | 31.30% | 4.09 |
| 5     | 38  | 22  | 11  | 71  | 329         | 65.8 | 11.37 | 2.776 | 14.11 | 21.45% | 4.15 |
| 6     | 35  | 18  | 9   | 62  | 391         | 65.2 | 10.28 | 2.571 | 10.79 | 16.56% | 4.12 |
| 7     | 39  | 23  | 11  | 73  | 464         | 66.3 | 9.84  | 2.447 | 9.10  | 13.74% | 4.17 |
| 8     | 28  | 19  | 11  | 58  | 522         | 65.3 | 9.57  | 2.365 | 8.00  | 12.27% | 4.12 |
| 9     | 29  | 23  | 10  | 62  | 584         | 64.9 | 9.02  | 2.306 | 6.93  | 10.69% | 4.11 |
| 10    | 33  | 23  | 13  | 69  | 653         | 65.3 | 8.60  | 2.262 | 6.15  | 9.42%  | 4.13 |

Table 2 shows the original data of average grain size determined by Planimetric Procedure. In Table 2, N_i is number of grains completely within the test circle, N_c is number of grains intercepted by the test circle.
circle, \( N_i \) is the total number of grains within the test circle on a metallographic photograph, and \( N = N_i + 0.5N_C \sum N_i \) is cumulative number of grains, \( N_V \) is average number of grains, and \( S_i \) is standard deviation of \( N_i \) within the test circle on several metallographic photographs. \( b_{95}\% (v) \) is the quantile of t-distribution under the n fields (degree of freedom \( v = n-1 \)) and confidence probability 95\%. 95\% CI is 95\% confidence interval of several \( N_V \). \%RA is percent relative accuracy under 95\% confidence probability, which is \( 95\% CI / N_V \). \( G \) is the ASTM grain size number obtained under \%RA limit of several metallographic photographs. According to the requirements of ASTM E112, when \%RA is less than or equal to 10\%, the \( G \) result is acceptable. After the thirteenth field has been detected, \( \sum N_{13} = 740 \), \( N_V = 56.9 \), 95\% RA = 9.44\% \leq 10\%, and \( G = 3.94 \). For the aluminium alloy (AlSi11) specimen, the ASTM grain size number is 3.94 determined by Planimetric Procedure.

### Table 2. The Planimetric Procedure results

| Field | \( N_i \) | \( N_C \) | \( \sum N_i \) | \( N_V \) | \( S_i \) | \( t_p (v) \) | 95\% CI | \%RA | \( G \) |
|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| 1     | 59    | 39    | 78.5  | 78.5  | 78.5  | -     | -     | -     | 4.40  |
| 2     | 38    | 33    | 54.5  | 133.0 | 66.5  | 16.97 | 12.706| 152.47| 229.28%| 4.16  |
| 3     | 29    | 23    | 40.5  | 173.5 | 57.8  | 19.22 | 4.303 | 47.74 | 82.55%| 3.96  |
| 4     | 38    | 29    | 52.5  | 226.0 | 56.5  | 15.92 | 3.182 | 25.32 | 44.82%| 3.92  |
| 5     | 40    | 31    | 55.5  | 281.5 | 56.3  | 13.79 | 2.776 | 17.12 | 30.41%| 3.92  |
| 6     | 44    | 29    | 58.5  | 340.0 | 56.7  | 12.37 | 2.571 | 12.98 | 22.91%| 3.93  |
| 7     | 51    | 27    | 64.5  | 404.5 | 57.8  | 11.67 | 2.447 | 10.80 | 18.68%| 3.96  |
| 8     | 30    | 36    | 48.0  | 452.5 | 56.6  | 11.35 | 2.365 | 9.49  | 16.77%| 3.93  |
| 9     | 37    | 32    | 53.0  | 505.5 | 56.2  | 10.68 | 2.306 | 8.21  | 14.62%| 3.92  |
| 10    | 44    | 32    | 60.0  | 565.5 | 56.6  | 10.14 | 2.262 | 7.25  | 12.83%| 3.93  |
| 11    | 46    | 29    | 60.5  | 626.0 | 56.9  | 9.69  | 2.228 | 6.51  | 11.44%| 3.94  |
| 12    | 40    | 30    | 55.0  | 681.0 | 56.8  | 9.26  | 2.201 | 5.88  | 10.37%| 3.93  |
| 13    | 39    | 40    | 59.0  | 740.0 | 56.9  | 8.89  | 2.179 | 5.37  | 9.44% | 3.94  |

### 4. Evaluation of measurement uncertainty

#### 4.1. Evaluation of uncertainty in parameter measurement

##### 4.1.1 Uncertainty evaluation of vernier caliper

Vernier calipers are used to measure the scale length and the diameter of test circle. According to GB/T 21390-2008 standard [6], the maximum permissible measurement error of vernier calipers with 0.02 mm scale is 0.03 mm when measuring length is less than 150 mm. The uncertainty caused by the vernier caliper is as follows:

\[
u_r = \sqrt{\left( \frac{0.03}{\sqrt{3}} \right)^2 + \left( \frac{0.02}{2\sqrt{3}} \right)^2} = 0.018
\]

##### 4.1.2 Uncertainty evaluation of magnification

The magnification of metallographic photograph is calculated from:

\[
M = \frac{D}{l}
\]

Where \( M \) is the magnification, \( l \) is scale length on the metallographic photograph \((l=200 \, \mu m=0.2 \, mm)\), and \( D \) is the measured result of \( l \). The \( D \) is determined twice, and the range \( R=0.02 mm \) and range coefficient \( C=1.13 \). The uncertainty caused by repeated measurement is as follows:

\[
u_r = \frac{R}{C} = \frac{0.02}{1.13} = 0.018
\]

The \( D \) is determined with the vernier calipers, the uncertainty caused by the vernier caliper has been described.
The combined uncertainty of $D$ measurement results is as follows:

$$u_{D-M} = \sqrt{u_i^2 + u_f^2} = \sqrt{0.018^2 + 0.018^2} = 0.025$$

Ignoring the uncertainty of $l$ itself, the uncertainty caused by magnification is as follows:

$$u_l = \frac{u_{D-M}}{0.2} = \frac{\sqrt{0.018^2 + 0.018^2}}{0.2} = \frac{0.025}{0.2} = 0.13$$

### 4.1.3 Uncertainty evaluation of test circle total circumference

The total circumference of three test circles is calculated from:

$$L = \pi d_1 + \pi d_2 + \pi d_3$$ (4)

Where $d_1$, $d_2$, and $d_3$ are diameters of three test circle in mm.

The horizontal and vertical diameters of each circle are measured with vernier calipers, and the range $R=0.02$ mm and range coefficient $C=1.13$. In order to avoid the correlation, three circle diameters are measured by different vernier calipers.

The uncertainty evaluation of $d_1$, $d_2$, and $d_3$ is the same as the uncertainty evaluation of $D$. And the uncertainty caused by $d_1$, $d_2$, and $d_3$ is as follows:

$$u_d = \sqrt{u_i^2 + u_f^2} = \sqrt{0.018^2 + 0.018^2} = 0.025$$

The combined uncertainty of test circle total circumference is as follows:

$$u_L = \sqrt{(\pi u_{d_1})^2 + (\pi u_{d_2})^2 + (\pi u_{d_3})^2} = \sqrt{3(\pi u_d)^2} = 0.14$$

### 4.1.4 Uncertainty evaluation of test circle area

The test circle area is calculated from:

$$A = \frac{1}{4} \pi d^2$$ (5)

Where $d$ is the diameter of test circle in mm.

The horizontal and vertical diameters of test circle are measured with vernier calipers, the average diameter is 79.86 mm, the range $R=0.02$ mm and range coefficient $C=1.13$. The uncertainty evaluation of $d$ is the same as the uncertainty evaluation of $D$. And the uncertainty caused by $d$ is as follows:

$$u_d = \sqrt{u_i^2 + u_f^2} = \sqrt{0.018^2 + 0.018^2} = 0.025$$

The combined uncertainty of test circle area is as follows:

$$u_A = \frac{\pi d}{2} = \frac{\pi \times 79.86 \times 0.025}{2} = 3.2$$

### 4.1.5 Uncertainty evaluation of repeated measurement

For Abrams Three-Circle Procedure, the G of the specimen was determined by using 10 fields, as shown in Table 1. For 10 fields, the average grain boundary intersection count is 65.3 and the standard deviation is 8.60, that is, $P=65.3$, $S_{10}=8.60$. The uncertainty caused by repeated $P$ measurement is as follows:

$$u_{P_1} = \frac{S_{10}}{\sqrt{n}} = \frac{8.60}{\sqrt{10}} = 2.7$$

According to the experience of statistical count, when an individual metallographer counts the grain boundary intersection count, the counting error of every field is ±2. The uncertainty caused by counting error is as follows:

$$u_{P_2} = \frac{2}{\sqrt{3}} = 1.2$$

The combined uncertainty of repeated measurement using Abrams Three-Circle Procedure is as follows:

$$u_P = \sqrt{u_{P_1}^2 + u_{P_2}^2} = \sqrt{2.7^2 + 1.2^2} = 3.0$$

For Planimetric Procedure, the G of the specimen was determined by using 13 fields, as shown in Table 2. For 13 fields, the average number of grains is 56.9 and the standard deviation of $N_{j}$ is 8.89, that is, $N_{p13}=56.9$, $S_{13}=8.89$. The uncertainty caused by repeated $N$ measurement is as follows:

$$u_{N_1} = \frac{S_{13}}{\sqrt{n}} = \frac{8.89}{\sqrt{13}} = 2.5$$
Also, according to the experience of statistical count, when an individual metallographer counts the number of grains, the counting error of every field is ±2. The uncertainty caused by counting error is as follows:

\[ u_{n2} = \frac{2}{\sqrt{3}} = 1.2 \]

The combined uncertainty of repeated measurement using Planimetric Procedure is as follows:

\[ u_N = \sqrt{u_{n1}^2 + u_{n2}^2} = \sqrt{2.5^2 + 1.2^2} = 2.7 \]

4.2. Evaluation of combined uncertainty

4.2.1 Abrams Three-Circle Procedure. For the aluminium alloy (AlSi1) specimen, the G is 4.13 determined by Abrams Three-Circle Procedure. In formula (1), \( M = 101.50 \times, L = 498.76 \text{ mm}, P = 65.3 \). And according to formula (1), the combined uncertainty of G is calculated from:

\[
G_{c-L-c} = \sqrt{\left( \frac{\partial G}{\partial M} \right)^2 u_M^2 + \left( \frac{\partial G}{\partial L} \right)^2 u_L^2 + \left( \frac{\partial G}{\partial P} \right)^2 u_P^2} \tag{6}
\]

Where:

\[ u_M = 0.13, \quad \frac{\partial G}{\partial M} = \frac{2.88539}{101.50} = 0.028 \]
\[ u_L = 0.14, \quad \frac{\partial G}{\partial L} = -\frac{2.88539}{498.76} = -0.0058 \]
\[ u_P = 3.0, \quad \frac{\partial G}{\partial P} = \frac{2.88539}{65.3} = 0.044 \]

The combined uncertainty is as follows:

\[ u_{G-L-c} = 0.131 \]

The expanded uncertainty are as follows:

\[ U = 0.26, \quad k=2 \]

The results of ASTM grain size number of aluminium alloy (AlSi1) specimen determined by Abrams Three-Circle Procedure are as follows:

\[ 4.13 \pm 0.26, \quad k=2 \]

4.2.2 Planimetric Procedure. For the aluminium alloy (AlSi1) specimen, the G is 3.94 determined by Planimetric Procedure. In formula (2), \( M = 101.50 \times, A = 5008.971 \text{ mm}^2, N = 56.9 \). And according to formula (2), the combined uncertainty of G is calculated from:

\[
G_{c-A-c} = \sqrt{\left( \frac{\partial G}{\partial M} \right)^2 u_M^2 + \left( \frac{\partial G}{\partial A} \right)^2 u_A^2 + \left( \frac{\partial G}{\partial N} \right)^2 u_N^2} \tag{7}
\]

Where:

\[ u_M = 0.13, \quad \frac{\partial G}{\partial M} = \frac{2.88539}{101.50} = 0.028 \]
\[ u_A = 3.2, \quad \frac{\partial G}{\partial A} = -\frac{1.442695}{5008.971} = -0.00029 \]
\[ u_N = 2.7, \quad \frac{\partial G}{\partial N} = \frac{1.442695}{56.9} = 0.025 \]

The combined uncertainty is as follows:

\[ u_{G-A-c} = 0.069 \]

The expanded uncertainty are as follows:

\[ U = 0.14, \quad k=2 \]

The results of ASTM grain size number of aluminium alloy (AlSi1) specimen determined by Planimetric Procedure are as follows:

\[ 3.94 \pm 0.14, \quad k=2 \]

5. Analysis and discussion
Using the Abrams Three-Circle Procedure determined \( G = 4.13 \), \( U = 0.26 \), \( k = 2 \); Using the Planimetric Procedure determined \( G = 3.94 \), \( U = 0.15 \), \( k = 2 \). The sources of uncertainty include: calipers for measuring circle diameter or scale length, magnification, three test circle total circumference, test circle area, errors of grain boundary intersection count or number of grains, repeated measurements.

In the table 3, \( U_P \) is the expanded uncertainty of \( G \) determined by the Abrams Three-Circle Procedure and \( U_N \) is the expanded uncertainty of \( G \) determined by the Planimetric Procedure.

Assuming that the permissible error of magnification changes from 0 to 2% and other conditions remain unchanged, the uncertainty caused by magnification is evaluated respectively according to uniform distribution and the uncertainty evaluation procedure in this paper. The \( U_P \) and \( U_N \) changing trends with the permissible error of magnification are shown in Table 3. In the table, \( \Delta R \) stands for magnification error.

Assuming that the scale of ruler changes from 0.01mm to 1mm, the maximum permissible measurement error of ruler meets JIG 30 standard \(^7\) or JIG 1 standard \(^8\), and other conditions remain unchanged, the uncertainty caused by length measurement is evaluated respectively according to uniform distribution and the uncertainty evaluation procedure in this paper. The \( U_P \) and \( U_N \) changing trends with the scale of ruler are shown in Table 3. In the table, CC stands for current calipers, SR stands for steel ruler, \( d \) stands for ruler scale, \( \Delta I \) stands for permissible measurement error.

Assuming that the error of statistical counting changes from 1 to 5 and other conditions remain unchanged, the uncertainty caused by statistical counting is evaluated respectively according to uniform distribution and the uncertainty evaluation procedure in this paper. The \( U_P \) and \( U_N \) changing trends with error of statistical counting are shown in Table 3. In the table, \( \Delta 2 \) stands for statistical counting.

Table 3. Sources of uncertainty and expanded uncertainty

| Magnification | Length measurement | Statistical counting |
|---------------|--------------------|----------------------|
| \( d \) | \( \Delta I \) | \( U_P \) | \( U_N \) | \( \Delta 3 \) | \( U_P \) | \( U_N \) |
| 0.0% | 0.26 | 0.14 | 0.01 | 0.03 | 0.26 | 0.14 | 1 | 0.25 | 0.13 |
| 0.5% | 0.26 | 0.14 | 0.02 | 0.03 | 0.26 | 0.14 | 2 | 0.26 | 0.14 |
| 1.0% | 0.26 | 0.14 | 0.05 | 0.05 | 0.26 | 0.14 | 3 | 0.29 | 0.15 |
| 1.5% | 0.27 | 0.14 | 0.10 | 0.10 | 0.26 | 0.14 | 4 | 0.32 | 0.17 |
| 2.0% | 0.27 | 0.15 | 0.1 | 0.1 | 0.28 | 0.17 | 5 | 0.35 | 0.19 |

The extended uncertainty is up to two significant digits, usually one significant digit. If the extended uncertainty is rounded off according to the 1/3 rule, the \( U_P \) is 0.3 and the \( U_N \) is 0.2. From Table 3, it can be seen that the expanded uncertainty does not change with the magnification error and the ruler scale. It can be concluded that the contributions of the uncertainty caused by the magnification, the ruler, the scale length or test circle circumference, and test circle area to the uncertainty of \( G \) determined by the Abrams Three-Circle Procedure or the Planimetric Procedure can be negligible, when the magnification (\( \leq 2\% \)) and the ruler (1mm scale) meet the requirements of ASTM E112 standard. It can be seen that the uncertainty of \( G \) is 0.02 different when the ruler scale is 1mm and 0.10 mm. In order to reduce the determination risk, it is suggested that the ASTM E112 standard should provide that the ruler that determines the diameter of test circles or scale length should be with the scale that is less than or equal to 0.10 mm.

According to the experience of statistical count, when an individual metallographer counts the grain boundary intersection count or number of grains, the counting error of every field is \( \pm 2 \). ASTM E112 also requires control of 40 to 100 grain boundary intersection counts or 50 to 100 numbers of grains of every field. The lower limit number is required to ensure the boundary conditions of the mathematical model for calculating \( G \), while the upper limit number is to ensure the accuracy of statistical counting and reduce the counting error. From Table 3, it can be seen that the expanded uncertainty changes greatly when the statistical counting error exceeds 2. So it is very important to control the counting error in the range of \( \leq 2 \). In order to reduce the determination risk, it is suggested that the ASTM E112 standard should provide that the errors of counting should be less than or equal to 2. And for that, the
following methods can be adopted: 1) using counter; 2) labelling grain boundary intersection count or number of grains recommended by ISO 643:2012 standard [9]; 3) displaying method of specimen grain boundary; 4) ensuring gray difference between measurement grid or test circle and grain boundary.

Through the above analysis and discussion, it can be seen that the uncertainty of G determined by the Abrams Three-Circle Procedure or the Planimetric Procedure mainly comes from the repeated measurement of different field on the specimen, which is consistent with the repeatability requirement of ASTM E112 for controlling the detection precision. Repeatability measurements actually reflect the uniformity of the specimen grain size. Selecting measurement field randomly, fairly and representatively, may increase the number of measurement field. But this selecting method can reduce measurement uncertainty, improve accuracy and reduce the detection risk.

In Table 1, the field number is 10 when %RA≤10%. Formula (1) is used to calculate G of every field, and the standard deviation of G is 0.39. That is to say, under the condition of meeting the requirement (%RA≤10%) of ASTM E112 standard, the precision of G measurement is not up to the standard requirement (±0.25). In Table 2, the field number is 13 when %RA≤10%. Formula (2) is used to calculate G of every field, and the standard deviation of G is 0.22. That is to say, under the condition of meeting the requirement (%RA≤10%) of ASTM E112 standard, the precision of G measurement is up to the standard requirement (±0.25). It can be seen that the %RA limit of ASTM E112 for determining G should be redefined according to the measurement uncertainty in order to be up to the precision requirement (±0.25).

6. Conclusion

In this paper, the ASTM grain size number of aluminium alloy specimen was determined respectively by Abrams Three–Circle Procedure and Planimetric Procedure of ASTM E112-13 Standard. The test pattern total lengths used in Abrams Three-Circle Procedure are 500 mm, and the test pattern areas used in Planimetric Procedure are 5000 mm². And the uncertainty is evaluated. The results are as follows:

1) Using the Abrams Three-Circle Procedure determines G, G=4.13, U=0.26, k=2; Using the Planimetric Procedure determines G, G=3.94, U=0.15, k=2.

2) It is suggested that the ASTM E112 standard should provide that the errors of counting grain boundary intersection count or numbers of grains should be less than or equal to 2, and the ruler that determines the diameter of test circle or scale length should be with the scale that is less than or equal to 0.10 mm. On this condition, the uncertainties caused by them in determining G can be negligible.

3) The main sources of uncertainty for the determination of G by Abrams Three-Circle Procedure and Planimetric Procedure are repetitive measurement, which depends on the uniformity of grain size of specimen.

4) The %RA limit of ASTM E112 for determining G should be redefined according to the measurement uncertainty in order to be up to the precision requirement (±0.25).

References

[1] ASTM E112-13 Standard Test Methods for Determining Average Grain Size[S].
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