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Acceptability analysis for determining the 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 by the Abrams three-circle procedure and planimetric procedure, according to ASTM E112-13 standard, and the measurement uncertainty was evaluated. Based on the test data and theoretical analysis, the %RA limit for acceptability of ASTM E112 grain size measurement was discussed. The following two corrections are made: when G is determined via the Abrams three-circle and planimetric procedures, the %RA limit should not exceed 8.4% and 16.1%, respectively, instead of earlier regulated value of 10%.

1. Introduction

The grain size of metallic materials directly affects the strength and plasticity of metallic materials and other specifications. Determining average grain size is a very important test item in the field of metallic materials testing. The standard ASTM E112-55 was originally approved in 1955 by ASTM, and the active version was approved in 2013, namely ASTM E112-13 standard test methods for determining average grain size [1]. Since its first publication, it has been revised more than 10 times for over 60 years.

ASTM E112-13 requires that in the statistical analysis of grain size, 95% confidence interval (95%CI) and percent relative accuracy (%RA) should be calculated based on the results of repetitive measurements. As a general rule, a 10% RA (or lower) is considered to be acceptable precision for most purposes. In this case, a precision of ± 0.25 grain size units can be attained, and results are free of bias.

According to the ISO/IEC 17025-2017 accreditation criteria for the competence of testing and calibration laboratories [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. While 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 metallic materials, uncertainty evaluation of measurement results is widely used. There are many published papers about the uncertainty evaluation and standardization has been achieved [3,4] in these two fields. However, in the field of metallographic examination, the uncertainty evaluation of measurement results is seldom used.



Bi et al. attempted for the first time to evaluate the uncertainty of grain size measurement results and draws the conclusions as follows: 1) It was 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. 2) The main sources of uncertainty for the determination of G by the Abrams three-circle procedure (ATCP) and planimetric procedure (PP) are repetitive measurement, which depends on the homogeneity of grain size of specimen. 3) 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) [5].

In this paper, the grain size of aluminum alloy specimen was determined by ATCP and PP of ASTM E112-13 Standard, and the uncertainty of the result was evaluated, in order to get the new %RA limit. On the basis of the test data, uncertainty and theoretical analysis, the new %RA limit for acceptability of ASTM E112 grain size measurement were discussed. The new %RA limit should ensure that the precision of ± 0.25 grain size units can be attained completely.

2. Testing scheme

The test specimen is aluminum alloy (AlSi1). The dimensions of polished surface are 18 mm x 10 mm. The film on the surface of specimen is prepared by anodization. On the whole polished surface, the grain sizes are observed and the images are captured under polarized light randomly without overlapping fields. The magnification of objective lens is 10.

The quantitative detection methods of G are as follows: 1) ATCP, the designed diameters of three test circles are 79.58 mm, 53.05 mm, and 26.53 mm. 2) PP, the designed diameter of test circle is 79.80 mm.

In the captured metallographic photographs, the scale and test circles are superimposed, and then the $100 \times$ photographs are printed. On the printed photographs, the scale length and diameter of test circles are measured, intersections of grain boundary and three test circles are counted, the number of grains completely within the test circle and the ones intercepted by the test circle are also counted.

The four vernier calipers with 0.02 mm scale are used to determine the scale length and test circle diameters. The scale length is measured twice and the diameters of the circles are measured twice in the horizontal and vertical directions. Then the average scale length and average diameters of circle are calculated. When counting grain boundary intersections or the number of grains, appropriate marks on the printed photographs are used to reduce the statistical counting error.

Via the ATCP, the ASTM-regulated G is calculated as follows:

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

where M is the magnification, L is the test circle total circumference in mm, and P is number of intersection counts.

Via the PP, the ASTM-regulated G is calculated as

$$G = 3.3219281 \lg \left(\frac{M^2}{A} N \right) - 2.954 \quad (2)$$

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

3. Test results

Figure 1 depicts a metallographic photograph superimposed with the three circles and with a scale of 200 μm length for the ATCP. Figure 2 presents a metallographic photograph superimposed with the circle and with a scale of 200 μm length for the PP.

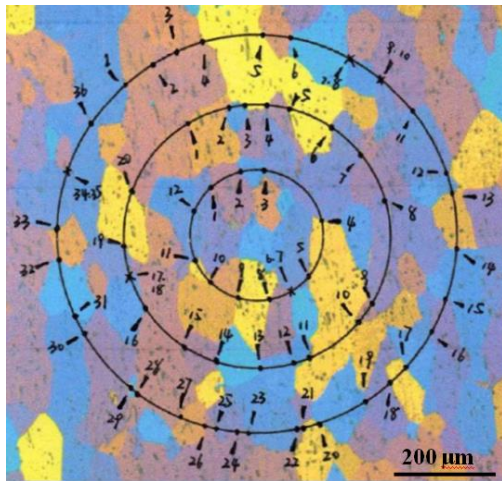


Figure 1. Three test circles and intersection mark.

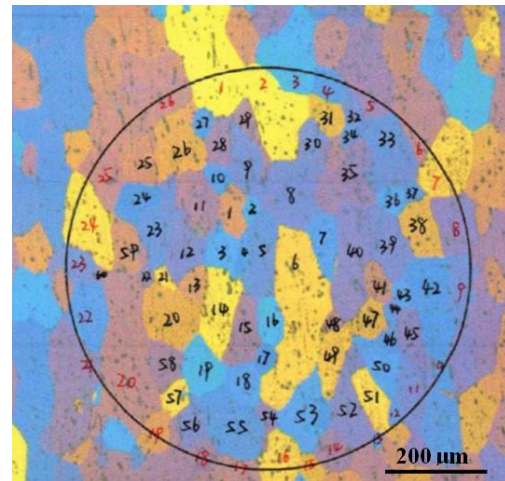


Figure 2. Test circle and grain number mark.

The average scale length in the metallographic photograph is 20.62mm. The actual magnification of metallographic photograph is $103.1\times$, that is, $M=103.1$ in formulas (1) and (2). The average diameters of the three circles in the metallographic photograph (see figure 1) are 79.67, 53.17, and 26.54 mm, respectively. The actual total circumference of the three circles is 500.72 mm, that is, $L=500.72$ mm in formula (1). The average circle diameter in the metallographic photograph (see figure 2) is 80.00 mm. The actual circle area is 5026.55 mm², that is, $A = 5026.55$ mm² in formula (2).

Table 1 shows the test data of average grain size number determined by ATPC. In table 1, P_i is the total grain boundary intersection count in one metallographic photograph (see figure1). P is the average grain boundary intersection count and $\sum P_i$ is the cumulative grain boundary intersection count in n metallographic photographs. Table 2 shows the test data of average grain size number determined by PP. In table 2, N_i is the total number of grains within the test circle in one metallographic photograph (see figure2). N is average number of grains and $\sum N_i$ is the cumulative number of grains in n metallographic photographs.

Table 1. The ATPC results.

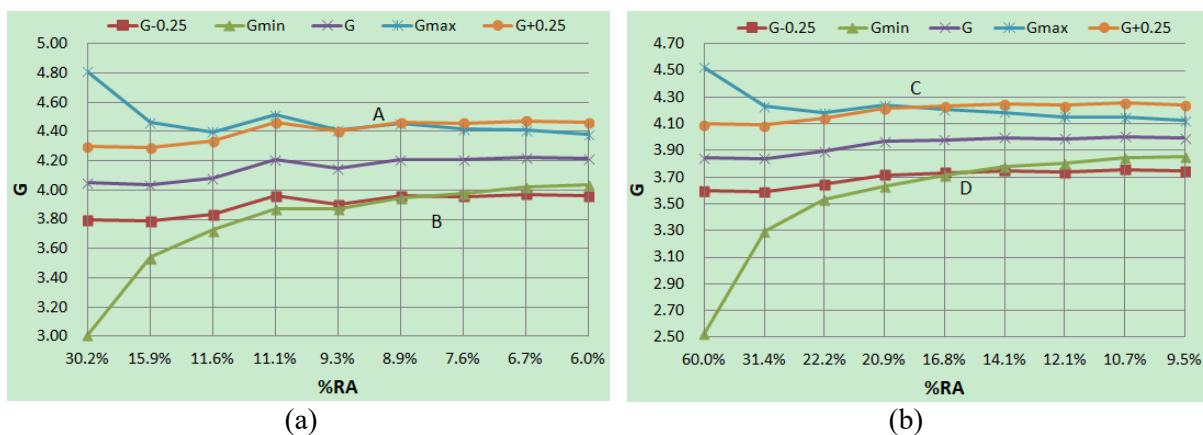
n	P_i	P	$\sum P$	95%CI	95%RA	G-0.25	Gmin	G	Gmax	G+0.25
1	62	62.00	62	-	-	3.81	-	4.06	-	4.31
2	54	58.00	116	50.82	87.63%	3.63	-	3.88	5.69	4.13
3	69	61.67	185	18.65	30.24%	3.80	3.01	4.05	4.81	4.30
4	61	61.50	246	9.76	15.88%	3.79	3.54	4.04	4.46	4.29
5	67	62.60	313	7.27	11.61%	3.83	3.73	4.08	4.40	4.33
6	74	65.38	523	7.27	11.12%	3.96	3.87	4.21	4.51	4.46
7	62	64.14	449	5.98	9.32%	3.90	3.87	4.15	4.41	4.40
8	74	65.38	523	5.79	8.86%	3.96	3.94	4.21	4.46	4.46
9	65	65.33	588	4.98	7.62%	3.96	3.98	4.21	4.42	4.46
10	68	65.60	656	4.41	6.73%	3.97	4.02	4.22	4.41	4.47
11	64	65.45	720	3.94	6.03%	3.96	4.03	4.21	4.38	4.46
12	69	65.75	789	3.62	5.50%	3.98	4.06	4.23	4.38	4.48
13	68	65.92	857	3.31	5.03%	3.99	4.09	4.24	4.38	4.49
14	70	66.21	927	3.11	4.69%	4.00	4.11	4.25	4.39	4.50

Table 2. The PP results.

n	Ni	N	ΣN	95%CI	95%RA	G-0.25	Gmin	G	Gmax	G+0.25
1	65.5	65.50	65.5	-	-	3.91	-	4.16	-	4.41
2	40.0	52.75	105.5	162.00	307.11%	3.60	-	3.85	5.87	4.10
3	53.0	52.83	158.5	31.68	59.96%	3.60	2.53	3.85	4.52	4.10
4	52.5	52.75	211.0	16.57	31.40%	3.59	3.30	3.84	4.23	4.09
5	61.0	54.40	272.0	12.10	22.23%	3.65	3.53	3.90	4.19	4.15
6	72.5	57.42	344.5	11.99	20.89%	3.72	3.63	3.97	4.24	4.22
7	62.0	58.07	406.5	9.78	16.84%	3.73	3.71	3.98	4.21	4.23
8	60.0	58.31	466.5	8.20	14.07%	3.75	3.78	4.00	4.19	4.25
9	57.0	58.17	523.5	7.06	12.14%	3.74	3.80	3.99	4.15	4.24
10	61.5	58.50	585.0	6.24	10.67%	3.76	3.84	4.01	4.15	4.26
11	59.5	58.59	644.5	5.57	9.50%	3.74	3.85	3.99	4.13	4.24
12	51.5	58.00	696.0	5.18	8.94%	3.74	3.85	3.99	4.11	4.24
13	73.0	59.15	769.0	5.35	9.04%	3.77	3.88	4.02	4.14	4.27
14	80.0	60.64	849.0	5.87	9.68%	3.79	3.90	4.04	4.18	4.29

In tables 1 and 2, n is the serial number of field, 95%CI is the 95% confidence interval of P or N . %RA is the percent relative accuracy under 95% confidence probability, that is $95\%CI / P$ or $95\%CI / N$. G is the ASTM grain size number obtained under the %RA limit of n metallographic photographs. G_{min} is a G value calculated from the lower limit of confidence interval of P or N ($P-95\%CI$ or $N-95\%CI$). G_{max} is a G value calculated from the upper limit of confidence interval of P or N ($P+95\%CI$ or $N+95\%CI$). ($G-0.25$) is the G value minus the precision of 0.25, ($G+0.25$) is the G value plus the precision of 0.25. ($G-0.25$) and ($G+0.25$) are equivalent to the lower and upper limit of the precision that the G can be attained by ASTM E112 standard. That is to say, for a %RA, the confidence interval of G determined by 95% CI of P or N is [G_{min} , G_{max}], and the precision interval of G , according to the precision of ASTM E112, is [$G-0.25$, $G+0.25$].

According to the data from the third field to the eleventh field in table 1 and from the third field to the eleventh 1 field in table 2, the curves of G , G_{min} , G_{max} , $G-0.25$, $G+0.25$ with %RA as abscissa and G as ordinate, are plotted respectively as shown in figure 3.

**Figure 3.** %RA— G curves. (a) ATCP and (b) PP.

In figure 3(a), the curve of $G+0.25$ and the curve of G_{max} have an intersection point A. When %RA is less than point A, $G_{max} < G+0.25$, and the curve of $G-0.25$ and the curve of G_{min} have an intersection point B. When %RA is less than point B, $G_{min} > G-0.25$. That is to say, When %RA is less than point

B, $[G_{\min}, G_{\max}] \in [G-0.25, G+0.25]$. The %RA value of point A and point B are between 7.5% and 9.3%.

In figure 3(b), the curve of $G+0.25$ and the curve of $G_{\max}$ have an intersection point C. When %RA is less than point C, $G_{\max} < G+0.25$, and the curve of $G-0.25$ and the curve of $G_{\min}$ have an intersection point D. When %RA is less than point D, $G_{\min} > G-0.25$, which means, when %RA is less than point D, $[G_{\min}, G_{\max}] \in [G-0.25, G+0.25]$. The %RA value of point C is between 16.8% and 20.9%, and the %RA value of point D is between 14.1% and 16.8%.

4. Evaluation of measurement uncertainty

4.1. The expanded uncertainty U_{95}

According to reference [5], the uncertainty of G measurement results is evaluated. When the scale of the ruler for measuring the circle diameter and metallographic photograph scale is less than or equal to 0.1 mm and the statistical counting error is less than 2, the uncertainty of magnification, test circle total circumference, test circle area and statistical counting error can be neglected. The uncertainty of G measurement results mainly comes from the uncertainty introduced by repeated measurement. The testing conditions of test specimen meet the above requirements. Uncertainty evaluation of G measurement results is based on Type-A evaluation of measurement uncertainty [6,7].

The number of repeated measurements is n . According to the Bessel formula method, the standard deviation of n measurement results is s , and the uncertainty of the mean value of n measurement results is as follow:

$$u_c = \frac{s}{\sqrt{n}} \quad (3)$$

The expanded uncertainty with a coverage probability of 95% is:

$$U_{95} = k_{95} u_c = t_{95}(v_{\text{eff}}) \frac{s}{\sqrt{n}} \quad (4)$$

Where k_{95} is the coverage factor with a coverage probability of 95%, v_{eff} is the effective degree of freedom, and $t_{95}(v_{\text{eff}})$ is the quantile of t -distribution under n fields (degree of freedom $v = n-1$) and coverage probability of 95%. $t_{95}(v_{\text{eff}})$ can be extracted from tables in the standards [7,8].

4.2. The coverage interval of G by ATCP

In table 1, when $n = 7$, %RA = $9.97 \leq 10\%$, which meets the acceptability requirements of ASTM E112, there are $P = 64.14$, $s = 6.47$, $t_{95}(6) = 2.447$, 95% CI = 5.98, $G = 4.15$, and the confidence interval of G determined by 95% CI is in the range of [3.87, 4.41], which is larger than that determined by 4.15 ± 0.25 (see table 1 and figure 3(a)). According to formula (4), the value U_{95} of P , which was introduced by ATCP-based repeated measurements, is as follows:

$$U_{95-P} = 2.447 \frac{6.47}{\sqrt{7}} = 5.98$$

Thus, the coverage interval of P with a probability of 95% is as follows:

$$[P - U_{95-P}, P + U_{95-P}] \rightarrow [64.14 - 5.98, 64.14 + 5.98] \rightarrow [58.16, 70.12]$$

By substituting 64.14, 58.16 and 70.12 into formula (1), respectively, the following results are obtained: $G = 4.16$, $G_{\min} = 3.88$, $G_{\max} = 4.41$. Thus, the $G = 4.16$ for test specimens, and the coverage interval with coverage probability of 95% is in the range of [3.88, 4.41]. This coverage interval coincides with that determined by 95% CI.

4.3. The coverage interval of G by PP

In table 2, when $n = 11$, $\%RA = 9.52 \leq 10\%$, which meets the acceptability requirements of ASTM E112, there are $N=58.59$, $s=8.28$, $t_{95}(10)=2.228$, $95\% CI = 5.57$, $G=3.99$, and the confidence interval of G determined by $95\% CI$ is in the range of $[3.85, 4.13]$. The confidence interval of G is in the range of 4.00 ± 0.25 (see table 2 and figure 3(b)). According to formula (4), the U_{95} value of N , which was introduced by repeated measurements via ATCP, is as follows:

$$U_{95-N} = 2.228 \frac{8.28}{\sqrt{11}} = 5.57$$

Thus, the coverage interval of N with a probability of 95% is:

$$[N - U_{95-P}, N + U_{95-P}] \rightarrow [58.59 - 5.57, 58.59 + 5.57] \rightarrow [53.03, 64.16]$$

By substituting 58.59 , 53.03 and 64.16 into formula (2), respectively, the results are as follows: $G=3.99$, $G_{min}=3.86$, $G_{max}=4.13$. Thus, the $G=3.99$ for test specimens, and the coverage interval with coverage probability of 95% is in the range of $[3.86, 4.13]$. This coverage interval is basically the same as that determined by $95\% CI$.

4.4. Confidence interval and coverage interval of G

ASTM E112 specifies the requirements of magnification and the ruler for measuring length [1]. In the case of meeting the requirements, the influence of magnification, the test circle total circumference and the test circle area on the test results is neglected, and the statistical analysis of grain size determination is used.

According to ASTM E112, the $95\% CI$ of P or N is derived as follows:

$$95\% CI = \frac{t \cdot s}{\sqrt{n}} \quad (5)$$

The calculation formula (5) for $95\% CI$ and the formula (4) for U_{95} are identical. Thus:

$$\%95 CI = U_{95} \quad (6)$$

It is concluded that the confidence interval of 95% confidence probability obtained by statistical techniques is the same as coverage interval of 95% coverage probability obtained by uncertainty evaluation. This conclusion holds for the previous test and calculated data. The theoretical analysis is consistent with the experimental data.

5. The $\%RA$ limit

5.1. The symmetric interval of G

The statistical calculation of ASTM E112 is based on P or N values, not on G , because G itself has no physical significance.

According to the $\%RA$ formula of ASTM E112 and formula (6), there is:

$$\%RA = \frac{95\% CI}{\bar{X}} = \frac{U_{95}}{\bar{X}} \quad (7)$$

According to the formula (7), for the ATCP, there is:

$$\%RA_p = \frac{95\% CI}{P} = \frac{U_{95-P}}{P} \quad (8)$$

According to the formula (7), for the PP, there is:

$$\%RA_N = \frac{95\% CI}{N} = \frac{U_{95-N}}{N} \quad (9)$$

According to formulas (1) and (2), when the uncertainties of M , L , and A are neglected, the combined

uncertainty of G is calculated from:

$$u_{G-L-c} = \sqrt{\left(\frac{\partial G}{\partial P}\right)^2 u_P^2} = \sqrt{\left(\frac{2.88539}{P}\right)^2 u_P^2} = 2.88539 \frac{u_P}{P} \quad (10)$$

$$u_{G-A-c} = \sqrt{\left(\frac{\partial G}{\partial N}\right)^2 u_N^2} = \sqrt{\left(\frac{1.442695}{N}\right)^2 u_N^2} = 1.442695 \frac{u_N}{N} \quad (11)$$

From formulas (10) and (11), the extended uncertainty with coverage probability of 95% can be further derived as follows:

$$U_{95-G-L} = 2.88539 \frac{U_{95-P}}{P} \quad (12)$$

$$U_{95-G-A} = 1.442695 \frac{U_{95-N}}{N} \quad (13)$$

According to formulas (8), (12), (19), and (13), it can be concluded that:

$$\%RA_P = 0.34657 U_{95-G-L} \quad (14)$$

$$\%RA_N = 0.69315 U_{95-G-A} \quad (15)$$

According to formulas (14) and (15), if $U_{95} \leq 0.25$, then $\%RA_P \leq 8.66\%$ or $\%RA_N \leq 17.33\%$. That is to say, for the ATCP, when $\%RA_P$ is less than or equal to 8.66%, the G has the (± 0.25) coverage interval or confidence interval with probability of 95%. For the PP, when $\%RA_N$ is less than or equal to 17.33%, the G has the (± 0.25) coverage interval or confidence interval with probability of 95%.

The main reason of the differences of $\%RA$ limit between two quantitative approaches are from the constant 6.6438576 of formula (1) and the constant 3.321928 of formula (2). This means that the sensitivity coefficients are nearly twice as different [5-7]. This difference has been shown in formulas (12) to (13) and formulas (14) to (15). In fact, if U_{95-G-A} is equal to U_{95-G-L} , according to formulas (14) and (15), it can be deduced that the $\%RA_P$ is equal to $0.5 \times \%RA_N$.

5.2. The asymmetric interval of G

Formulas (1) and (2) are logarithmic functions. When the input is a symmetric interval, the output is an asymmetric interval. That is to say, if the input is P in the symmetric interval $[P-U_{95-P}, P+U_{95-P}]$ or N in the symmetric interval $[N-U_{95-N}, N+U_{95-N}]$, the resulting G interval is not a symmetric interval $[G-U_{95}, G+U_{95}]$, but an asymmetric interval $[G_{min}, G_{max}]$.

For the ATCP, by substituting P , U_{95-P} , $P-U_{95-P}$, $P+U_{95-P}$ into formula (1), it can be deduced that:

$$G_{max} - G = 6.643856 \lg(1 + \%RA_P) \quad (16)$$

$$G - G_{min} = 6.643856 \lg \frac{1}{1 - \%RA_P} \quad (17)$$

According to formula (16), if $G_{max} - G \leq 0.25$, then $\%RA_P \leq 8.87\%$. This corresponds to point A in figure 3(a). According to formula (17), if $G - G_{min} \leq 0.25$, then $\%RA_P \leq 8.44\%$. This corresponds to point B in figure 3(a). It can be seen that only when $\%RA_P$ is less than or equal to 8.44%, $[G_{min}, G_{max}] \in [G - 0.25, G + 0.25]$. The theoretical values are in good agreement with the test data, as is seen in figure 3(a).

For the PP, by substituting N , U_{95-N} , $N-U_{95-N}$, $N+U_{95-N}$ into formula (2), it can be deduced that:

$$G_{max} - G = 3.321928(1 + \%RA_N) \quad (18)$$

$$G - G_{min} = 3.321928 \lg \frac{1}{1 - \%RA_N} \quad (19)$$

According to formula (18), if $G_{\max}-G \leq 0.25$, then $\%RA_N \leq 19.33\%$. This corresponds to point C in figure 3(b). According to formula (19), if $G-G_{\min} \leq 0.25$, then $\%RA_N \leq 16.17\%$. This corresponds to point D in figure 3(b). It can be seen that only when $\%RA_N$ is less than or equal to 16.17%, $[G_{\min}, G_{\max}] \in [G-0.25, G+0.25]$. The theoretical values are in good agreement with the test data, as is seen in figure 3(b).

It can be seen that, in order to ensure that the G coverage interval or confidence interval with probability of 95% is in the range of $G \pm 0.25$, the %RA limit should not exceed 8.44% for the ATCP and 16.17% for the PP. These conclusions are different from those deduced from formulas (14) and (15) that the %RA limit should not exceed 8.66% or 17.33% for the ATCP and PP, respectively. This difference comes from formulas (1) and (2). According to formulas (16), (17), (18), and (19), the conclusions are in line with the reality.

Because the uncertainty of magnification, test circle total circumference, test circle area and statistical counting error are neglected and in order to reduce the measurement risk, ASTM E112 is suggested to specify that: the %RA limit should be less than or equal to 8.4% for the ATCP and the %RA limit should be less than or equal to 16.1% for the PP. The new %RA limits could ensure that the precision of ± 0.25 grain size units can be attained completely.

When the acceptability of G is evaluated with $\%RA \leq 10\%$, $\%RA_P \leq 8.66\%$, and $\%RA_P \leq 8.4\%$, respectively, for the ATCP, the corresponding results of G are shown in tables 3 (see table 1 and figure 3(a)). It can be seen that the decrease of the %RA limit leads to the increase of the field number, but ensures that the confidence interval and coverage interval is within the precision range of (± 0.25) and reduces the measurement risk.

When the acceptability of G is evaluated with $\%RA \leq 10\%$, $\%RA \leq 17.33\%$, and $\%RA \leq 16.1\%$, respectively, for the PP, the corresponding results of G are shown in tables 3 (see table 1 and figure 3(b)). It can be seen that the confidence interval or coverage interval is enlarged in the precision range of (± 0.25) due to the increase of the %RA limit, but the field number is reduced and the measurement efficiency is improved.

Table 3. The %RA limit comparative results.

Quantitative method	ATCP			PP		
%RA limit	$\leq 10\%$	$\leq 8.86\%$	$\leq 8.4\%$	$\leq 10\%$	$\leq 16.1\%$	$\leq 17.33\%$
%RA test result	9.32%	8.86%	7.62%	9.50%	14.07%	16.9%
N	7	8	9	11	8	7
G	4.15	4.21	4.21	3.99	4.00	3.98
$[G_{\min}, G_{\max}]$	[3.87,4.41]	[3.94,4.46]	[3.98,4.42]	[3.85,4.13]	[3.78,4.19]	[3.71,4.21]
$[G-0.25, G+0.25]$	[3.90,4.46]	[3.96,4.46]	[3.96,4.46]	[3.74,4.24]	[3.75,4.25]	[3.73,4.23]

6. Conclusion

Based on the test data and theoretical analysis of ASTM grain size number of aluminium alloy specimen, the new %RA limit for acceptability of ASTM E112 grain size measurement was discussed. The results are as follows:

- When using the ATCP to determine G, the %RA limit should not exceed 8.4%, instead of 10%. Because of the decrease of %RA limit, the field number is increased, but the measurement risk is reduced by ensuring that the confidence interval or coverage interval is within the precision range of (± 0.25) .
- When using the PP to determine G, the %RA limit should not exceed 16.1% instead of 10%. Because of the increase of %RA limit, the field number is reduced and the measurement efficiency is improved.

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