Effect of Observed Number of Inclusions on the Diameter Distribution at Two-Dimensional Inspection

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The effects of observed number of inclusions on the diameter distribution at two-dimensional inspection were evaluated in this work. Microscopic observations were reproduced by numerical calculations. Numerically calculated diameters of circles at cross-sectional planes were aggregated as size and density histograms and those were compared with theoretically calculated histograms. Deviations between the calculated and true density in each class were evaluated quantitatively with the error evaluation index.

The reliability of two-dimensional particle size and density histogram was affected by the observed number of particles rather than the observed number of fields and independent of the input three-dimensional particle distribution. Misunderstandings in reading the histograms could be prevented by setting small number of classes when the observed number of particles is small. It is better to decide the goal of reliability level before inspection to optimize the observation time and cost.

KEY WORDS: inclusions; histogram; distribution; microscope; two-dimensional inspection.

1. Introduction

The mechanical properties of steel are strongly affected by the nonmetallic inclusion. Many trials had applied to improve these properties by not only controlling the compositions of steel, but also the compositions, size, shape and distribution of inclusion. For example, in the case of the free cutting steel, which is used for machined products, their surface roughness and chip disposability are improved by dispersing low aspect ratio manganese sulfide uniformly.1,2) In the case of the steel plate, which is used for welded structural fabrication, their strength and toughness of heat-affected zone are improved by dispersing fine inclusion to prevent the grain growth as the pinning effect.3) On the other hand, in the case of the bearing steel, which is used in long time, heavy loaded and severe conditions, inclusion is thoroughly eliminated to improve their rolling contact fatigue life. As mentioned above, innovative methods to control inclusion finely and precisely are required and this demand would become strong in the future.

For the observation method of inclusion, the direct inspection on well polished metal surface with optical microscope is the conventional method. The geometrical characteristics of inclusion, such as size, shape and number, are evaluated by a skillful operator and the histogram of the size and frequency distribution of observed inclusion is shown for the comparison of cleanliness. As well as the optical microscope, these characteristics are observed with the scanning electron microscope and the compositions of inclusion are simultaneously is because the formation path and phase of inclusion might be considered in case of shape control of inclusion.

The quantitative evaluation method of its distribution is defined in ISO 4967 and JIS G0555. These methods are widely used for delivery inspection by comparing distribution of inclusion in 200 mm² with standard diagram. The advantage of these direct observation methods is that the distribution of inclusion in steel could be clarified directly. On the other hand, the disadvantage is that the true diameter and shape of inclusion are never found due to the two-dimensional inspection. Therefore, the acid or non-aqueous solvent extraction method is applied for inclusion evaluation and the inclusions are filtered on the membranous filter to observe the three-dimensional diameter and shape of inclusions.4) These geometrical characteristics could be evaluated by the filtration method, however, the original distribution of inclusion in steel, which is related to the various properties, would be lost. Furthermore, extraction solvent must be selected carefully in order to avoid the dissolution of inclusion itself. From these points, the direct observation on well polished surface with the optical microscope would still be the major observation method if the analytical device would have improved.

The clean steel, such as bearing steel, is strongly affected by the presence of inclusion. Owing to the progress of steel refining technology, the concentration of total oxygen, which indicates the cleanliness of the steel, reached several mass ppm so that the size and frequency of appearance of inclusion at the observation is considerably reduced. On the observation of inclusion, at the moment, the most important criterion for it is field number, in other words, observed
area. Not only the expansion of observed area, but also the high resolution of microscopy and the automatic measurement system would be required when the cleanliness of clean steel was evaluated. However, it is difficult to detect enough inclusions in order to evaluate the cleanliness because the amount of inclusions would be decreased more and more. Thus, uncertainty of the size distribution would be brought by the lack of observed inclusions. On the basis of clean steel production, the size distribution of inclusion is one of the important factors and is required for the enough reliability.

These points were discussed elsewhere such as “Quantitative Microscopy”65 and some previous researchers revealed the effect of increasing observation area on the reliability of inclusion evaluation6 and the effect of classification width of the size on it.7 However, the relationship between the reliability of size distribution and the observed number of inclusions by microscopic observation may not have been referred to in previous works. Taking into consideration the recent progress of cleanliness of steel and the reduction of the frequency of the appearance, the importance of these discussions would be raised.

Therefore, calculation method, which can reproduce the trend of size distribution of inclusion with increasing the field number at two-dimensional inspection, was investigated. By comparing the numerically and theoretically calculated two-dimensional particle diameter and density histogram with the error evaluation index, the effect of the observed number of inclusions on diameter distributions was discussed in this paper.

2. Experimental Method

2.1. Numerical Calculation

The schematic illustration of numerical thought experiment is shown in Fig. 1. Spherical particles, which have three-dimensional (3-D) size distributions, are virtually dispersed in a 1 mm³ cubic. The cross-sectional plane is set in the cubic and the diameters of the circles which appeared on the plane are calculated numerically. The calculation condition is shown in Table 1. Diameters of particles are set up to 10.0 μm and their coordinates are given randomly. Actually, inclusions over 10.0 μm are sometimes found at an actual inspection but these probabilities are so small that particles over 10.0 μm are not discussed in this work. Probabilities of large size inclusions have to be evaluated with an extreme value distribution.8

The distribution of the circle diameters on the cross-sectional planes can be calculated by referring their original 3-D particle diameters, coordinates and the height of cross-sectional plane when the cubic is cut horizontally. By changing the height of cross-sectional plane or settings of input 3-D particle size distributions, microscopic observations can be numerically reproduced. Two-dimensional size and density histogram of particles on the cross-sectional planes (2-D histogram) is obtained by aggregating the calculated size and number of circles with calculated areas. Here, clustering and wrapping of the particles are not considered in this model. The particles located at the boundary of cubic wall are treated as same as the other particles in consideration of that the cubic is larger than the particle. Furthermore, input particle distribution is given as a histogram and a distribution in each class are set evenly.

2.2. Theoretical Calculation

The true size and density distribution of circles on the cross-sectional planes is calculated theoretically. The schematic illustration of the theoretical calculation is shown in Fig. 2. The probability of particle appearance on the cross-sectional plane is expressed as Eq. (1). The probability, where the diameter of circle \( D_{2D} \) would be classified into \( d_i – \Delta d \) to \( d_i \) class, is given by Eq. (2). Both equations are applied to all particles in the cubic and the theoretical 2-D histogram is obtained.

\[
P_c = \frac{\Delta d}{H} \text{.......................... (1)}
\]

\[
P_d = \frac{2 \sqrt{\frac{D_{2D}}{2}} - \left(\frac{d_i - \Delta d}{2}\right)^2 - 2 \sqrt{\frac{D_{2D}}{2}} - \left(\frac{d_i}{2}\right)^2}{D_{3D}} \text{...... (2)}
\]

\( P_c \): Probability of particle appearance on cross-sectional plane (–)

\( D_{3D} \): Diameter of 3-D particle (μm)

\( H \): Height of cubic (μm)

\( P_d \): Probability which the diameter of circle \( D_{2D} \) would be classified into \( d_i – \Delta d \) to \( d_i \) class (–)

\( d_i \): Threshold of class (μm)

\( \Delta d \): Classification (width of class) (μm)

2.3. Evaluation Method

Error evaluation index \( (I_{err}) \) is defined as Eq. (3) to evaluate the reliability of 2-D histogram by comparing quantitatively the difference between numerically and theoretically calculated 2-D histograms. The average of the relative deviation.

Table 1. Properties of the particles in numerical thought experiment.

| Item                | Setting       |
|---------------------|---------------|
| Shape               | Spherical     |
| Distribution        | Optional      |
| Diameter range      | 0–10.0 μm     |
| Volume              | Optional      |
| Number of class     | 100, 50, 20, 10, 5 |
Fig. 2. Schematic illustration of theoretical calculation.

The situations were simulated where 3-D particles representing aluminum oxide in steel, were dispersed in 1 mm³ cubic. Generally, the distribution of 3-D particle is affected by the generation, agglomeration and removal rate of particles and can be expressed as a lognormal distribution defined as Eq. (5). Three cases of input 3-D particle settings are shown in Table 2 and original distributions are shown in Fig. 3. Here, fast generation and removal rate is shown in case1, fast agglomeration and removal rate is shown in case2 and slow removal rate is shown in case3. The maximum particle diameter was set at 10.0 μm considering the diameter range of aluminum oxide in clean steel at an actual inspection. These original distributions were adjusted for the volume of aluminum oxide particles, which includes 5.0 mass ppm oxygen, and input 3-D particle distributions were obtained. Here, densities of aluminum oxide and steel were set at 3 980 kg/m³ and 7 700 kg/m³, respectively. Numerical calculations were tried three times in each case. Relations between field number, \( I_{err} \) and \( I_{inf} \) were discussed by changing the settings of input 3-D particle distribution.

\[
I_{err} = \sum_{i=1}^{k_{class}} \frac{\rho_{Cn} - \rho_{Tn}}{\rho_{Tn}} k_{class} + \varepsilon_{err} \quad \cdots \cdots \cdots (3)
\]

\[
I_{inf} = \sum_{i=1}^{k_{class}} \left(1 - \frac{\rho_{Cn} - \rho_{Tn}}{\rho_{Tn}} \right) + \varepsilon_{inf} \quad \cdots \cdots \cdots (4)
\]

As well as \( I_{err} \), information evaluation index (\( I_{inf} \)) is defined as Eq. (4) to evaluate the information of 2-D histogram quantitatively. \( I_{inf} \) would be high when \( k_{class} \) is large and \( I_{err} \) was enough small. The 2-D histogram with large \( I_{inf} \) is meaning that further information is involved, in another words, the histogram is approximately showing the true distribution. In Eq. (4), \( \varepsilon_{inf} \) is the correction number, which included the summation of all errors in the excess range. As same as \( \varepsilon_{err} \), \( \varepsilon_{inf} \) in a range over 10.0 μm is fixed to zero in this work.

\[
f(x, \mu, \sigma) = \frac{1}{\sqrt{2\pi\sigma^2}x} \exp\left\{-\frac{(\ln x - \mu)^2}{2\sigma^2}\right\} \quad \cdots \cdots (5)
\]

\( \mu \): Mean(–)
\( \sigma \): Standard deviation(–)

Table 2. Properties of the input 3-D particle.

| Item | Setting |
|------|---------|
| Volume /mm³ | Case1 | Case2 | Case3 |
| Density3D /mm³ | 2.05 x 10⁻⁴ ([O]=5.0 mass ppm as Al₂O₃) | 825 | 333 | 235 |
| Density3D /mm³ | 0.86 | 1.51 | 1.56 |
| \( \sigma \) | 0.60 | 0.20 | 0.40 |

Theoretical density3D /mm³ | 2.23 | 1.57 | 1.15 |

Input 3-D particle distribution in case1 is shown in Fig. 4. Numerically and theoretically calculated 2-D histograms in case1 are shown in Fig. 5. \( I_{err} \), which is indicated in the degree of reliability were smaller when increasing the field numbers during the calculation. Both numerical and theoretical results came closer together as the increasing field numbers so that decrease of \( I_{err} \) and increase of \( I_{inf} \) were found. Here, a range over 8.0 μm was not considered in case2 because the probability at this range was very small.

Relating to a particle shape, inclusions were assumed to be spherical particles in this work, but oval and rectangular inclusions are sometimes found at an actual inspection. Dispersion in a particle distribution would be widened when the particle shape is changed from a sphere so that an additional observed number of 2-D particles would be needed to reach the same reliability level. On the other hand, the reliability at an inspection is strongly affected by the cluster shape particles. There are only a few evaluation methods of cluster shape particle⁹ so that the discussion related to such complex shape particles is a future work.
3.2. Effect of Observed Number of Particles on Reliability

Relation between observed number of particles in the evaluated area \( N_{2D} \) and \( \text{I}_{\text{err}} \) is shown in Fig. 6. Linear relation on \( \text{I}_{\text{err}} \) was found in \( N_{2D} \) rather than a field number. In addition, small \( \text{I}_{\text{err}} \) could be found when the \( k_{\text{class}} \) was small at the same \( N_{2D} \) condition. Regarding these results, the reliability of 2-D histogram was strongly affected by both \( N_{2D} \) and a setting of \( k_{\text{class}} \). Furthermore, the slopes between \( N_{2D} \) and \( \text{I}_{\text{err}} \) were shown constantly around –0.48 regardless of \( k_{\text{class}} \) and their input 3-D particle distribution. Thus, enough \( N_{2D} \) would be required to improve \( \text{I}_{\text{err}} \), the reliability of 2-D histogram, in any input 3-D particle distribution, in other words, cleanliness conditions of the steel. However, four times \( N_{2D} \) are required to improve \( \text{I}_{\text{err}} \) half without changing \( k_{\text{class}} \) so that the goal of reliability level has to be decided before inspection to optimize the observation time and cost.

3.3. Effect of Number of Class on the Reliability

Relation between \( N_{2D} \) and \( \text{I}_{\text{inf}} \) in case1 is shown in Fig. 7. \( k_{\text{class}} \) was changed from 100 to 5 (classification was changed from 0.1 to 2.0 \( \mu \text{m} \)) so that the effect of \( k_{\text{class}} \) on the reliability of 2-D histogram could be investigated. An asymptotic curve forward \( k_{\text{class}} \) could be drawn with \( \text{I}_{\text{inf}} \) as increasing \( N_{2D} \). In addition, large \( N_{2D} \) was required to reach large \( \text{I}_{\text{inf}} \) at large \( k_{\text{class}} \) setting. On the other hand, \( \text{I}_{\text{inf}} \) at small \( k_{\text{class}} \) setting reached \( k_{\text{class}} \) readily even in small \( N_{2D} \). As shown in Fig. 6, \( \text{I}_{\text{err}} \) at large \( k_{\text{class}} \) was large and relative deviations in all class were summarized into \( \text{I}_{\text{inf}} \) so that dispersion of \( \text{I}_{\text{inf}} \) at large \( k_{\text{class}} \) setting was rather large. Similar trends were also found in case2 and case3.

Regarding these results, further information can be included in a 2-D histogram when \( k_{\text{class}} \) and \( N_{2D} \) are large. On the other hand, poor information can be included when \( N_{2D} \) is small and \( k_{\text{class}} \) is large. Needless to say, the 2-D histogram with infinite \( k_{\text{class}} \) and \( N_{2D} \) is ideal to clarify the particle distribution, but realistically \( N_{2D} \) is limited by the observation time and cost. Taking into consideration the
actual observation, the 2-D histogram with large $k_{\text{class}}$ is nonsense so that $k_{\text{class}}$ has to be set adequately as referring to $N_{2D}$. Large $k_{\text{class}}$ is sometimes set to make the particle distribution clearly. At that setting, however, both $I_{\text{inf}}$ and $I_{\text{err}}$ are small when $N_{2D}$ is small so that incomplete 2-D histogram would be drawn. If $k_{\text{class}}$ is set smaller, the maximum of $I_{\text{inf}}$ is reduced but not only $I_{\text{inf}}$ but also $I_{\text{err}}$ could be raised in the same $N_{2D}$ condition so that the reliability of 2-D histogram would be improved. Thus, misunderstandings in evaluating the particle density distribution from these 2-D histograms can be prevented to set $k_{\text{class}}$ adequately.

4. Conclusions

Calculation method which can reproduce the trend of particle size distribution with increasing the field number at microscopic inspection was developed. By comparing quantitatively the difference between numerically and theoretically calculated two-dimensional particle size and density histogram with the error evaluation index, the effect of the observed number of particles on diameter distributions was discussed in this work. The conclusions are as follows:

(1) The reliability of two-dimensional particle size and density histogram was affected by the observed number of particles rather than the observed field number and independent of the input three-dimensional particle distribution.

(2) Misunderstandings in reading the histogram could be prevented to set small number of classes when observed number of particles is small.

(3) The goal of reliability level has to be decided before inspection to optimize the observation time and cost.

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