

Practical Measurement of Fiber Particle Content in Titanate by Dynamic Image Analysis

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INTRODUCTION

To reduce WHO fiber* content in titanate such as $K_2Ti_6O_{13}$, $Na_2Ti_6O_{13}$ etc., one of the brake pad additives, correct measurement method with industrially practical methods is needed.

* ; major axis $\geq 5\mu m$, minor axis $\leq 3\mu m$, aspect ratio ≥ 3

Measurement using SEM and optical microscope [1][2] :

- Not practical since it takes quite a long time and very costly
- Not always accurate
- Large data scatter

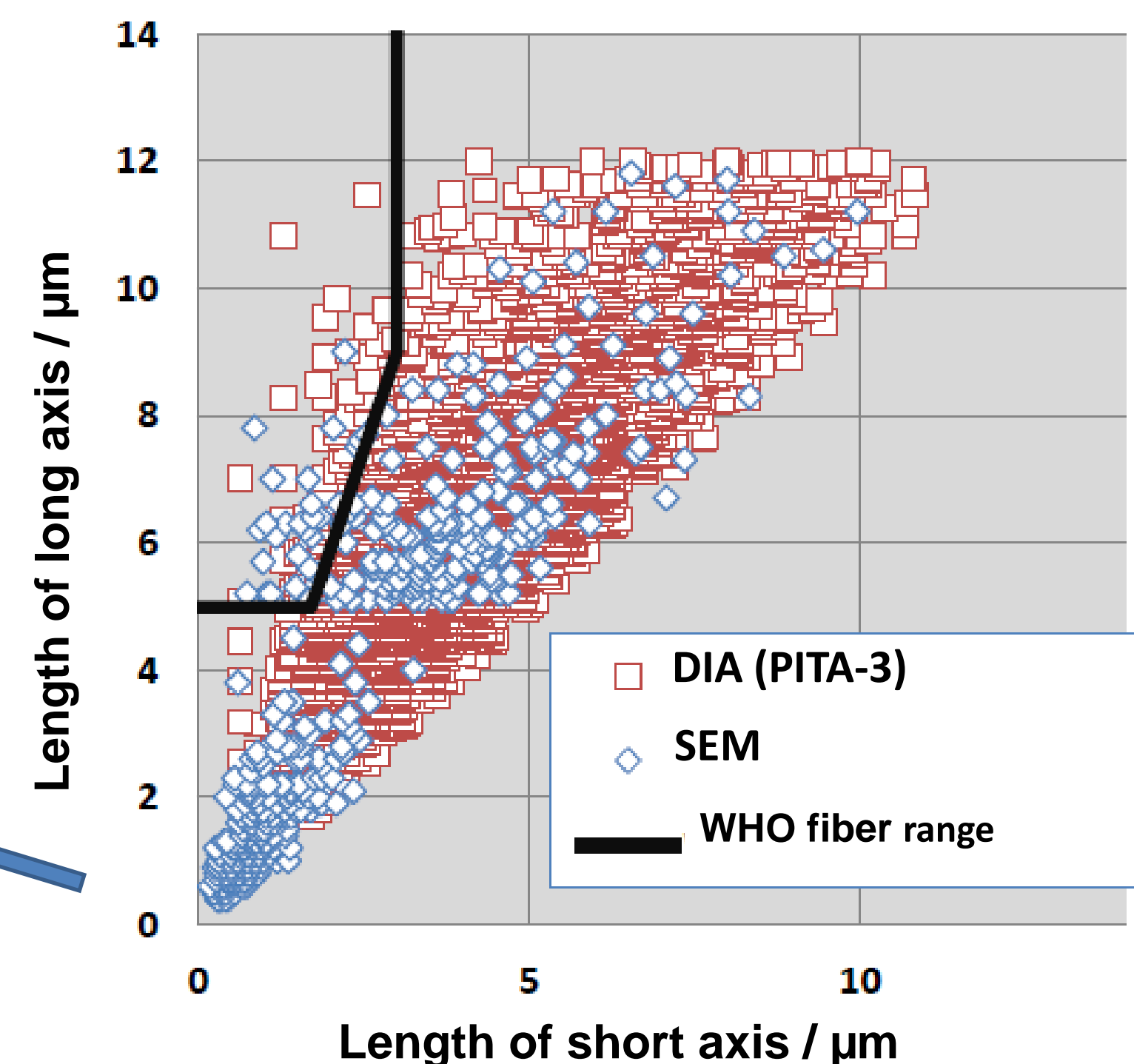
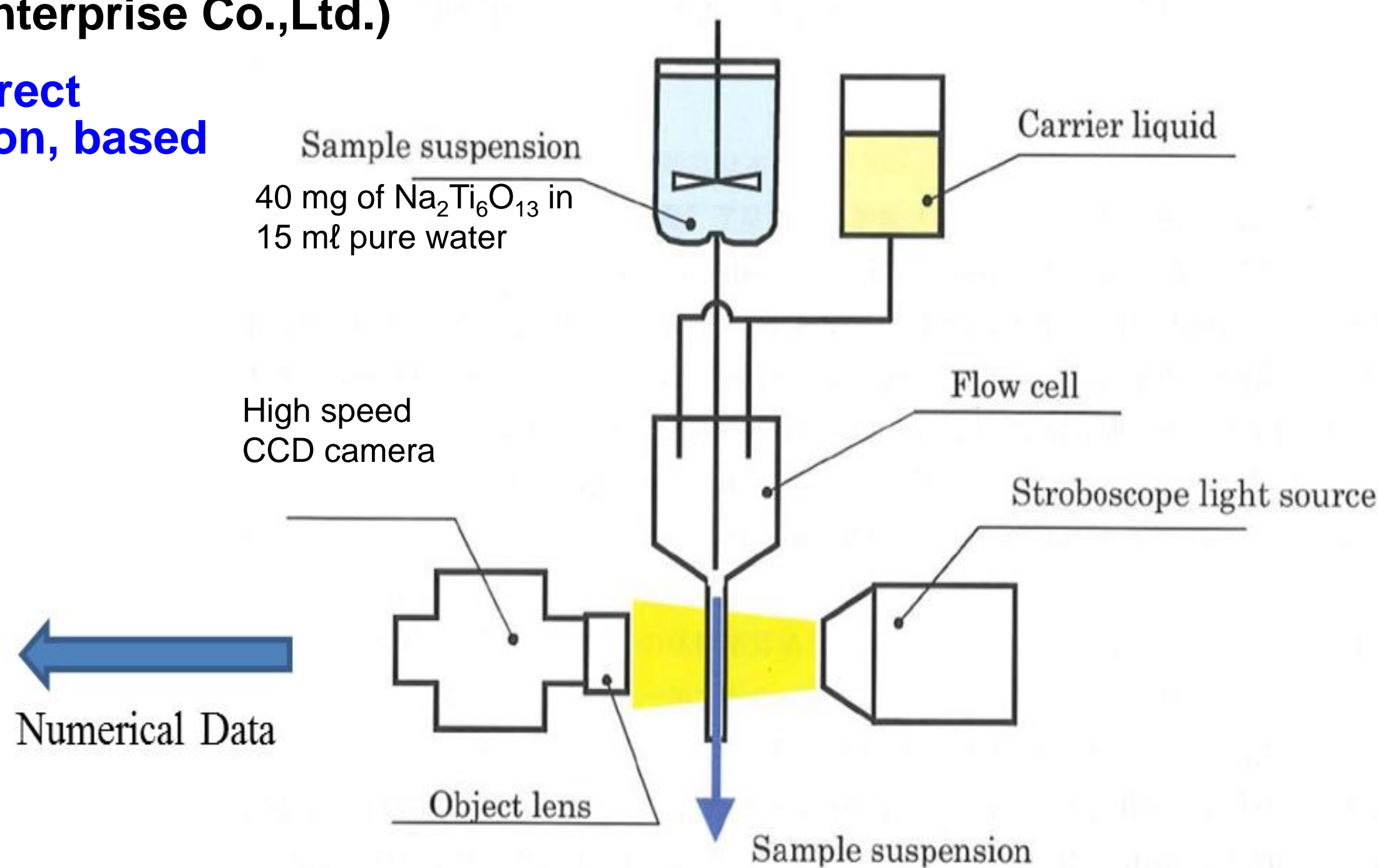
Our recommendation - Dynamic Image Analysis (DIA) [3]

- Around 70,000 particles in one product lot can be analyzed only in 10 min
- Fiber content value with very low data scatter
- Analytical results for fine particles smaller than $20\mu m$ well agree with the results by direct SEM observation.

PITA-3 (Seishin Enterprise Co.,Ltd.)

computer aided **direct optical observation, based on ISO 13322-2**

Computer :
Data storage
and analysis



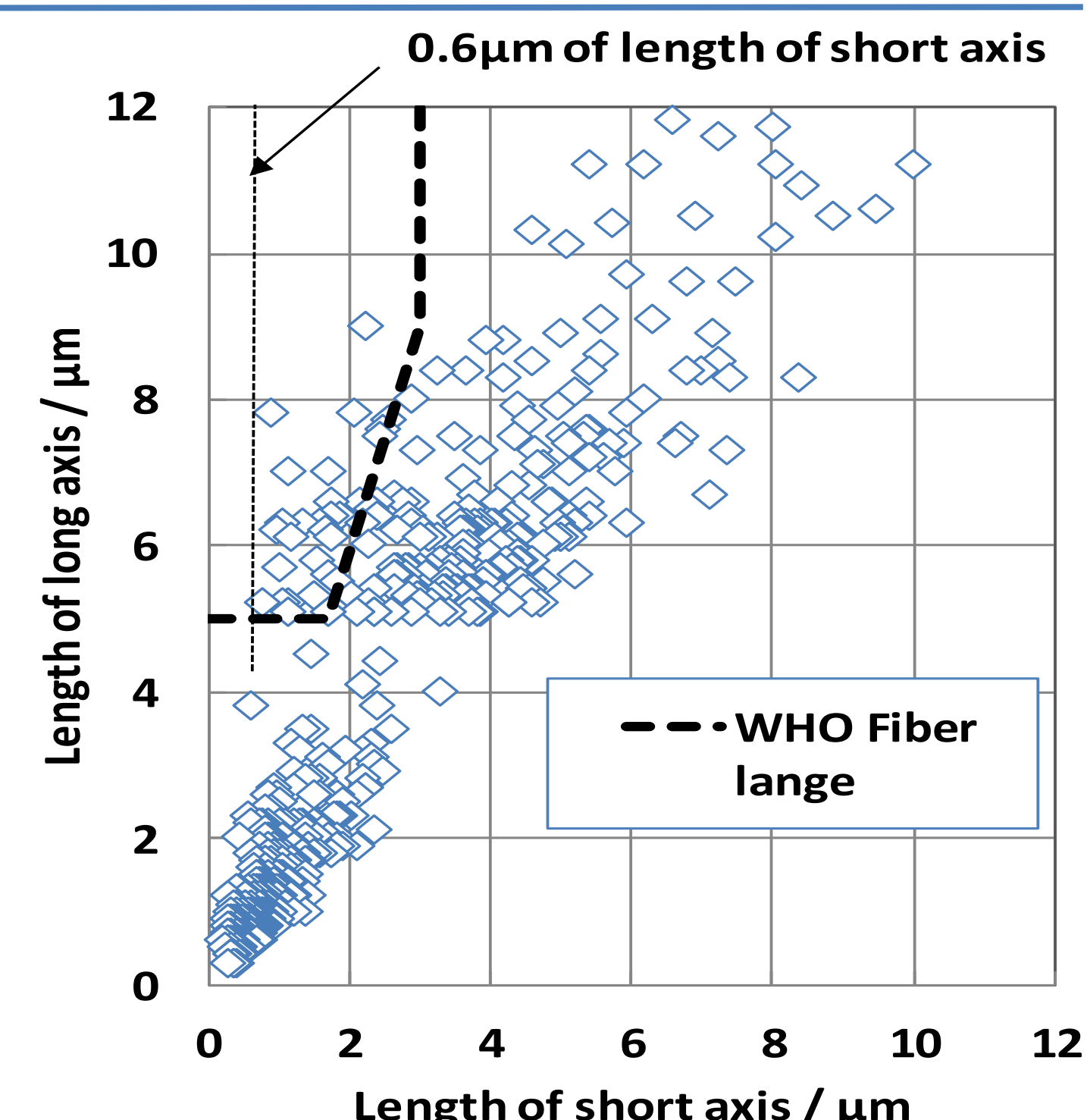
Comparison of DIA and SEM methods for TOHO's $Na_2Ti_6O_{13}$ [3]. Size distributions of particles smaller than $12\mu m$ are drawn.

- [1] World Health Organization, 1997, Determination of airborne fibre number concentrations, a recommended method by phase-contrast optical microscopy
- [2] Fraunhofer ITEM, 2018, Report to Toho Titanium "Measurement of the mass fraction of WHO fibers"
- [3] Taki, D., Sakai, H. and Fujii, H., 2019, Comparison of measurement methods for fiber particle content in titanate, Proc. of Eurobrake2019, EB2019-MDS-022

NEWLY CONDUCTED STUDY

Investigation on issues and attentions in the measurement of fiber particle contents by DIA

1. Resolution of the optical method can be improved by the use of an objective lens with higher magnification and higher resolution CCD camera.
2. However, small particles which cannot be clearly recognized by an optical method ($0.6\mu m$ of length of short axis) are all non-WHO fibers, which can be demonstrated by SEM observation, and DIA using visible light is enough for evaluating WHO fiber content in the titanate products investigated in this study.
3. By using the shape factor defined as the ratio of envelope peripheral length and peripheral length, some irregular particles such as overlapped ones in the photo images and aggregates / agglomerates can be effectively excluded for the estimation of WHO fiber content.



Size distributions of particles smaller than $12\mu m$ in TOHO's $Na_2Ti_6O_{13}$, observed with SEM. The data are the same ones in the upper figure.

CONCLUSION

DIA method is believed to be one of the best fiber content measurement methods for titanate from viewpoints of accuracy and practicality in spite of an optical method.