application note

Characterization of air-sensitive catalyst particles using the Morphologi® G3S particle characterization system in a glove box

Introduction

Supported Ziegler-Natta catalysts, such as those discovered by Kaminsky and Sinn in the early 1980's [1-3], are commonly used in polymer processing for olefin polymerization. The size of the catalysts is often measured as part of the Quality Control procedure during their manufacture. The shape of such supported catalysts is also of interested as it has been found to be related to shape of a growing polymer during the polymerization process.

Since Ziegler-Natta catalysts are sensitive to the air they must be analyzed in an inert atmosphere Previously, Scanning Electron Microscope (SEM) techniques have been used to analyze the size and shape of catalysts particles, however, since it is only possible to measure a limited number of particles by this technique the results are not statistically representative of the whole sample and therefore only limited information about size and shape distributions of the sample can be obtained.

The Morphologi G3S automated particle characterization system provides microscope quality images and delivers statistically significant data through the rapid analysis of thousands of particles with little or no user intervention giving significant savings in time and labor compared to analysis by SEM. The Morphologi G3S system can be conveniently housed inside a glove box therefore allowing analysis of the air sensitive catalyst particles in a controlled environment (Figure 1).

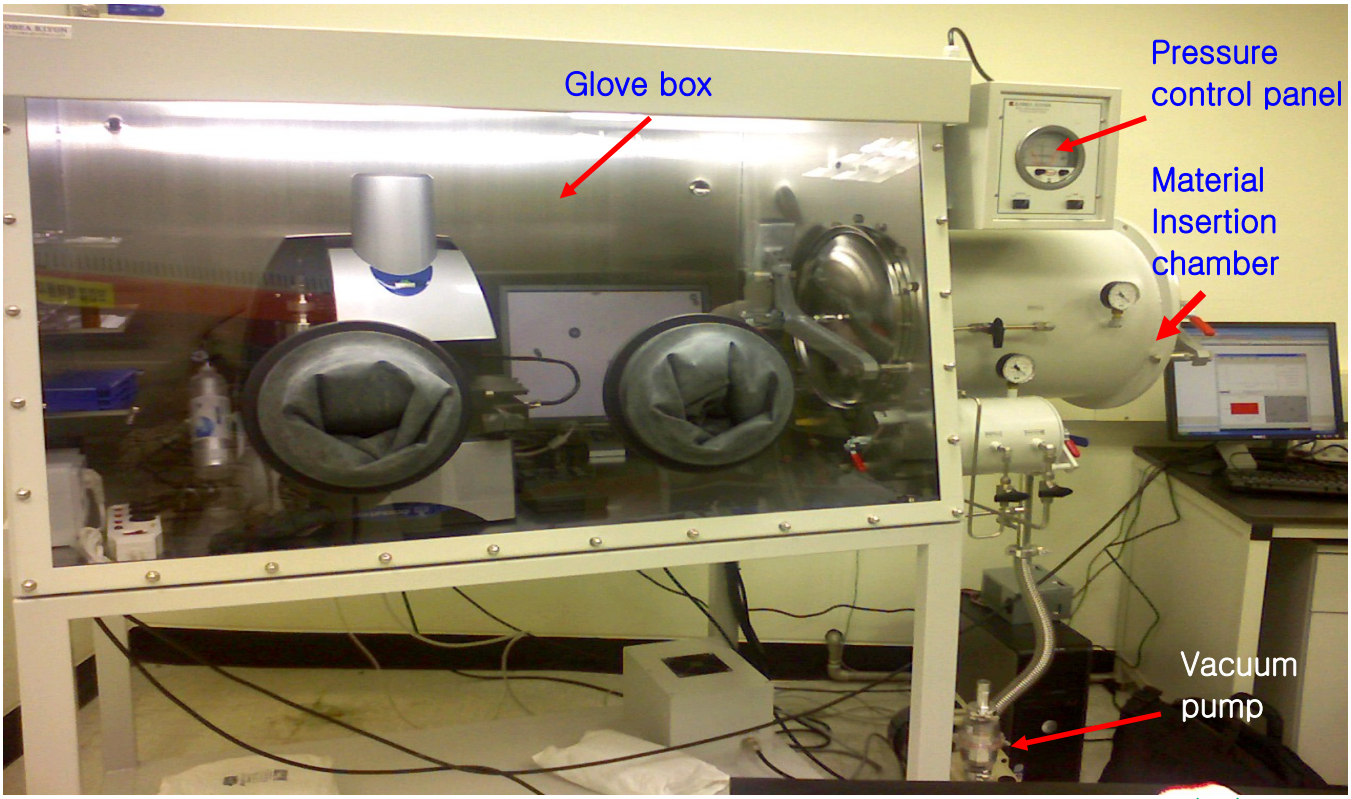

Figure 1: Morphologi G3 in the glove box system

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Example Experiment

Most supported Ziegler-Natta catalysts react to air, so analysis on the Morphologi G3S needs to be carried out in a glove box system to prevent the samples from exposure to air (Figure 1). The glove box system consists of the glove box itself, a material insertion chamber, a pressure control panel, a vacuum pump and a nitrogen purging system.

Before operating the instrument, all of the apparatus required for sample preparation, such as the clean Sample Dispersion Unit (SDU), glass plate, sample carrier etc. are placed into the glove box along with the sample itself via the material insertion chamber (Figure 2).

The air in the glove box is then replaced by nitrogen to provide an inert atmosphere.

For this example a Ziegler-Natta $MgCl_2$ supported catalyst sample was

Figure 2: Sample inserting part

analyzed. It is possible to tell if the particles have been exposed to air as they exhibit globules at their edges as shown in Figure 3.

Once the chamber was in the inert condition, the sample was dispersed using the integrated Sample Dispersion Unit (SDU) on the Morphologi G3S and was analyzed according to a Standard Operating Procedure (SOP) which defined all the software and hardware variables. Software filters were applied to remove all particle images containing less than 100 pixels from the analysis, to ensure good quality shape information was obtained, and to remove images of touching particles.

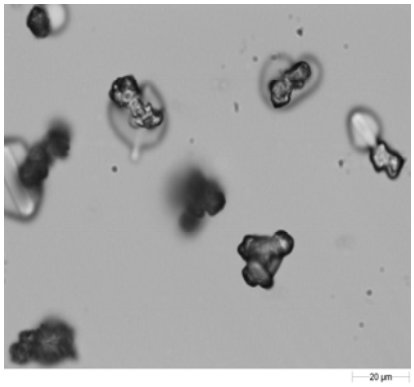


Figure 3: Field of view image of particles exposed to air

Results

The dispersion and analysis of more than 46000 particles took less than 15 minutes. Table 1 shows a summary of results in terms of size both on a number basis and a volume basis. The number based size distribution is shown in figure 4.

Figure 5 shows the HS Circularity distribution for the sample along with example images from near the two extremes of the distribution.

Table 1: Summary of size results by number and volume (µm)

mean	10.80		
D[n,0.1]	7.57	D[v,0.1]	9.04
D[n,0.5]	10.00	D[v,0.5]	12.91
D[n,0.9]	14.40	D[v,0.9]	20.27

Summary

The Morphologi G3S particle characterization system contained within a glove box provides an effective automated method to analyze air-sensitive Ziegler-Natta catalyst particles in statistically significant numbers. Results in terms of size and shape distributions are provided which are used for quality control and process monitoring during olefin polymerization.

References

[1] Korean J. Chem. Eng., 16(5) (1999), 562

[2] Korean J. Chem. Eng., 17(2) (2000), 205

[3] Korean J. Chem. Eng., 19)4) (2002), 557

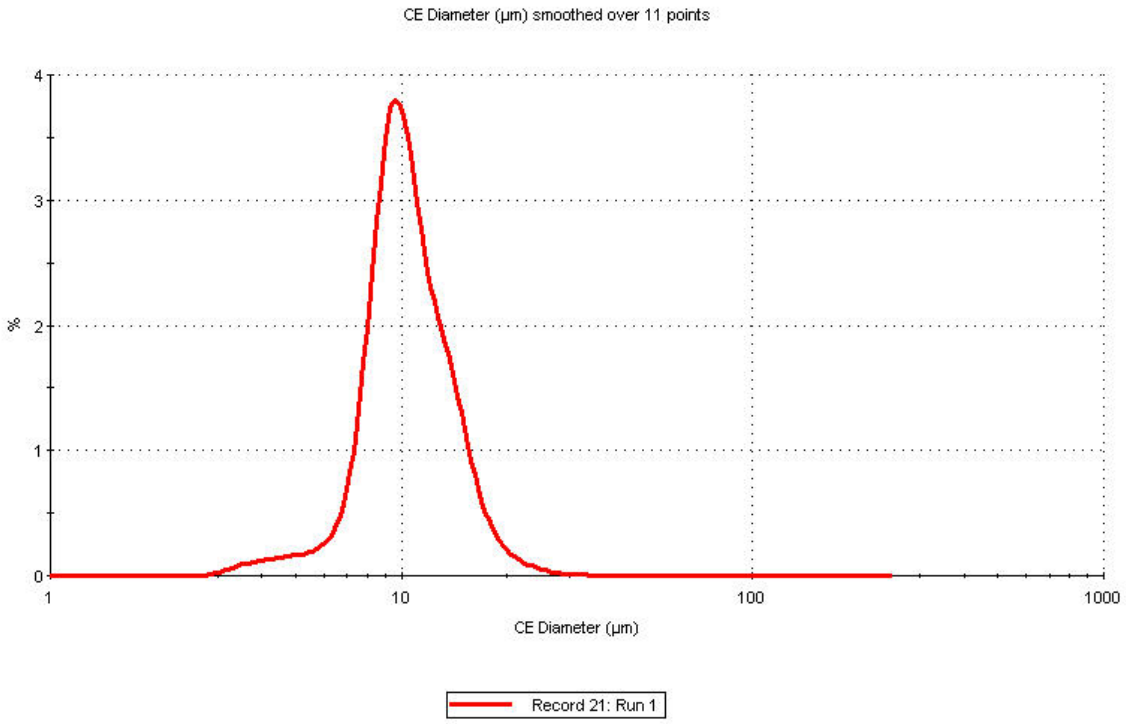


Figure 4: CE diameter distribution result for the analysis of the air sensitive Ziegler-Natta catalyst particles.

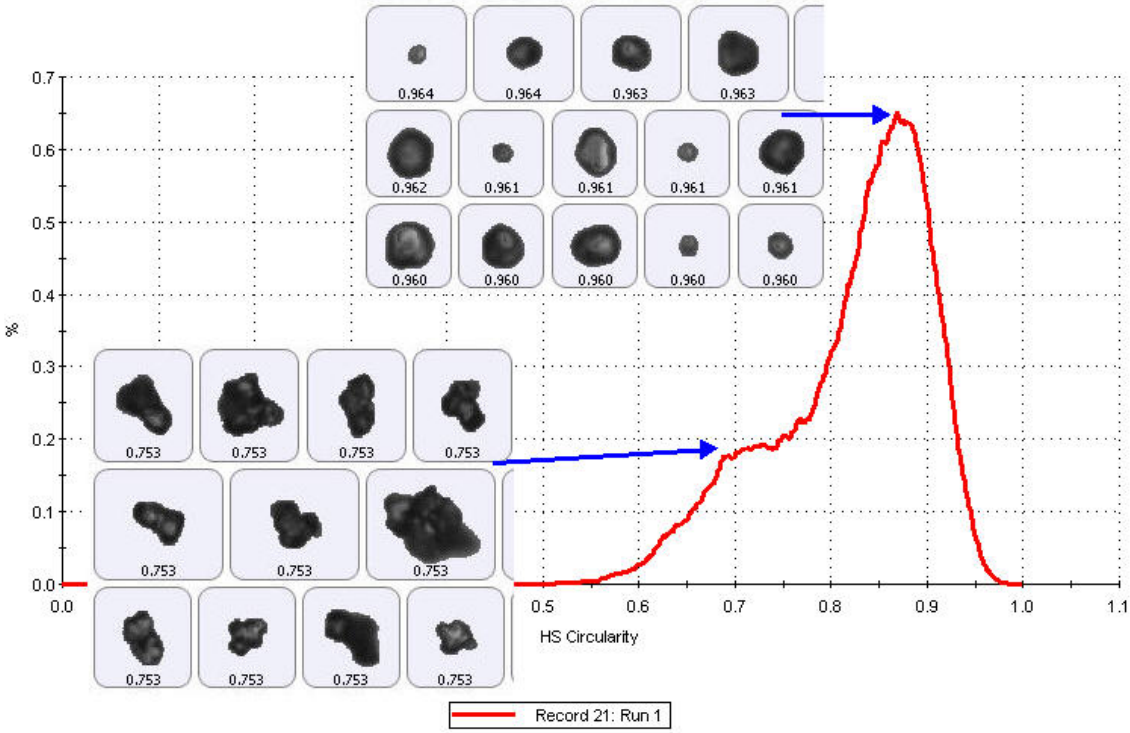
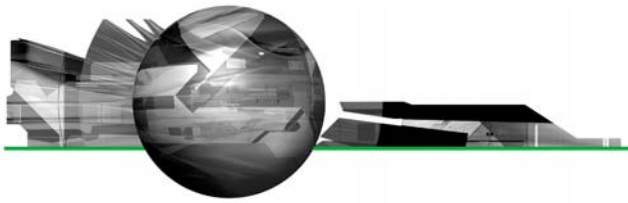


Figure 5: HS circularity distribution and images of more and less circular particles.



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