Large Particle Reduction by Porous Membrane Filter
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Introduction
Filtration has been contributing to keeping the number of micro scratches low and stable in CMP processes. Recent trends of lower concentration and smaller size of abrasive particles have enabled adoption of porous membrane filters for further micro scratch reduction with acceptable service life. Determining the large particle counts (LPC) of filtrate is a common practice for screening optimum filters before evaluating them at an actual CMP process. Conventional counting methods, which cover only particles larger than 0.5 µm size, could not show the difference in LPC among such fine grade filters. A new type particle counter and a control membrane test were used to differentiate porous membrane filtration relative to conventional melt-bonded depth filtration in terms of slurry filtrate quality.

Materials
Colloidal silica slurry
D50 = 20 nm, solid conc. = 20 mass%

Test filters
Depth filter A: Representative of fine grade melt-bonded depth filter
Membrane filter B: N66 porous membrane filter, 0.2 µm rated

Particle counter
AccuNo*1 FX-Nano (1 Trade mark of PSS)
Particle size distribution (PSD) analyzer
Zetasizer*2 Nano ZS (2 Trade mark of Malvern)

Filtration apparatus
Accurate dispersion by Bead mill

Method
Filtration of colloidal silica slurry
The slurry was filtered by the test filters at constant flow rate, 100 g/min to collect 1 kg of filtrate.

Large particle count
Large particles above 0.2 µm of the filtrates and the original slurry were measured by particle counter.

Control membrane test
For cross check, the filtrates and original slurry were filtered by 0.6 µm track etched membrane filter as control membrane at constant flow rate, 3 g/min. Pressure drop and the throughput were measured during filtration as shown in Fig. 3. Pressure drop increase in constant flow rate filtration is considered to be an indicator of LPC of the slurry.

PSD analysis
To confirm no removal of abrasive particles of acceptable size by filtration, particle size distribution of the filtrates and original slurry was measured by PSD analyzer.

Results and discussion
Significant LPC reduction of the filtrates was seen in Fig.2. The filtrate of depth filter A showed about 10% higher LPC than that of membrane filter B above 0.2 µm size. However, the difference was too small to confirm by only this result that membrane filter B provided better performance in LPC reduction.

Pressure drop increase of the control membrane in filtration of the filtrates and original slurry is compared in Fig. 3. Significant difference between the filtrates and original slurry was seen. The difference was large enough to establish better performance of membrane filter B in LPC reduction.

Combination of LPC above 0.2 µm and control membrane test demonstrated LPC reduction performance of porous membrane filter B.

PSD of the filtrates and the original slurry is confirmed to be no difference among them. The results indicated that necessary silica particles in original slurry passed the filters.

Influence of large particles on pressure drop increase of control membrane

Objective
To demonstrate “control membrane test” can detect small amount of large particles.

Materials
Colloidal silica slurry
The same slurry in the previous test.
PSD analyzer, filtration apparatus
The same apparatus in the previous test.

Method
Filtration, large particle count, PSD analysis
The same way as the previous test.

Control membrane test
The same way as the previous test except slurry samples were diluted 2 times by DIW.

Membrane filter B showed too rapid pressure drop increase. The filtrate of filter B was not studied further.

LPC above 0.2 µm of filtrates of filters A, C and original slurry is shown in Fig. 4. The filtrate of membrane filter C showed about 6% LPC reduction of original slurry. The difference was too small to indicate by only this result that membrane filter C would contribute to LPC reduction.

Pressure drop increase of control membrane in filtration of the filtrates and original slurry is compared in Fig. 5. Significant difference between the filtrate of membrane filter C and original slurry was seen. Combination of LPC above 0.2 µm and control membrane test indicated that porous membrane filter C would be effective to reduce LPC of the ceria slurry.

Results and discussion
PSD of the filtrates and the original slurry is confirmed to be no difference among them. The results indicated that necessary silica particles in original slurry passed the filters.

Conclusion
CMP slurries were filtered by porous membrane filters and a melt-bonded depth filter. LPC reduction of the filtrates was estimated by a particle counter in conjunction with control membrane test.

The results demonstrated that a combination of particle counter and control membrane test could confirm high performance of porous membrane filter for LPC reduction.

References

1. AccuNo*1 FX-Nano (Trade mark of PSS)
2. Zetasizer*2 Nano ZS (Trade mark of Malvern)