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# The Importance of Particle Size to the Performance of Abrasive Particle in the CMP Process

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Michael C. Pohl*, Duncan A. Griffiths*

## 1. Introduction

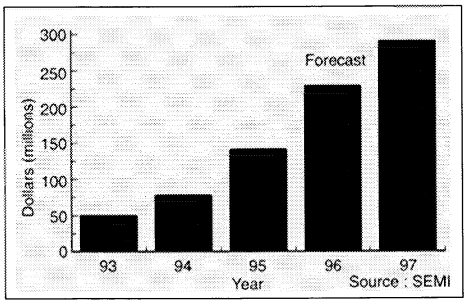

Fig.1 Consensus market forecast for CMP polishers

CMP is an area of technology which is growing at a very rapid pace. As Figure 1[1] shows, it is projected to continue its phenomenal growth over the next five years. In spite of its growth, it is still a relatively new field of investigation[2]. The mechanism of the process is understood to include oxidation and abrasion, but the details are a bit vague. The notion of applying an abrasive slurry to the wafer was quite radical and was expected to create some problems. One of the problems uncovered was the formation of micro-scratches in the wafer[3]. From other applications for abrasives this was known to be caused by the presence of larger particles in the slurry[4]. This problem suggested that particle size would be a critical area for study[5]. This idea has been borne out by some recent experiments.

## 2. Abrasive materials for CMP slurries

A wide variety of particulate materials have been used in abrasive processes to planerize solid surfaces[6]. These include $Al_2O_3$, $SiO_2$, $SiC_2$, $Si_3N_4$, diamonds and a host of other industrially important materials. In an abrasive procedure, the particle size is critical to controlling the rate of removal of the undesirable material. The problem is that at the point of total removal of the unwanted material, the etching must be halted very quickly. This need for etch rate control has led

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## ＣＭＰにおける研磨材の特性に及ぼす粒子径の重要性

### 1. はじめに

CMP技術は成長著しい分野ではあるが(図1)，研磨機構の詳細は未解明である．半導体プロセスにおける問題の一つにウエハに生じる微細なキズがあるが，これはスラリ中に存在する大きな粒子が原因だとされている．

### 2. CMP用スラリの研磨材

CMP用スラリには$Al_2O_3$, $SiO_2$, $SiC_2$, $Si_3N_4$, ダイアモンドなどの微粒子が研磨材として使われており，その粒径は不純物除去制御上で重要因子である．未発表だが，微細なキズとスラリ中の大きな粒子との間には密接な関係があるという研究もある．米国政府仕様#C16195Aは粒径別に研磨材を指定しており，CMP用スラリにも同じ分類が適用されている．

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to extensive studies of the abrasive particles. Unpublished work has established a strong correlation between micro-scratching and the presence of stray larger particles. This knowledge has led to abrasives being specified by size according to U.S. Government Specification #C16195A. This same sort of classification has been applied to CMP slurries7).

## 3. Particle size distribution analysis of the abrasive materials

A variety of techniques are available to size the abrasive particles used in the CMP slurries8). Far and away the fastest growing and most widely used is laser light scattering9). Most estimates have sales of these products growing at rates in excess of 15% per year. Its speed, ease of use, ready application to process control and other characteristics suggest its use to study CMP slurries. One can readily envision its use to study slurries immediately before and after contact with the wafer surface. Study of process operating parameters as well as checking incoming raw materials seem very useful applications. The information which could be obtained is very extensive.

The theory of operation of the laser light scattering instruments is very simple10). Basically, different sized particles scatter light at different angles. If you measure the intensity of the light at a series of different angles, you can calculate the particle size distribution. At this point it becomes interesting because many different algorithms exist to calculate the particle size distribution. As a result, different results can be determined for the same sample, depending on the algorithm selected. Horiba has chosen to use a non-linear iterative regression analysis to solve this problem11). This selection has proven valuable in a number of well documented cases12).

## 4. Measured data

One key area of validation is in the factory QA procedures performed on every instrument13). Examples of this type of analysis are shown in Figures 2 and 3. These quartz and poly-styrene samples are considered to be valid standards by the particle sizing community. The close correspondence of the median size, 0.411 μ m for the 0.405 μ m PSL and the 1.080 μ m for the 1.150 μ m quartz suggest the accuracy of this instrument. Several overlays for the same two samples are presented in Figures 4 and 5. The close agreement in both cases document

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## 3. 研磨材の粒径分布の測定

スラリ中の研磨材の粒径分布測定にはいくつかの方法があるが，中でも，レーザ光散乱式が最も広く使われており，測定装置の売上げは年率15%以上の伸びが見込まれている．本分析装置は分析速度が速くて使い易いなどの特長があり，スラリの研究開発はもちろん，受入れ検査やCMPプロセス・パラメータの研究など，さまざまな用途が検討されている．

レーザ光散乱式の測定原理は単純で，粒子の大きさによる光の散乱角度の違い，つまり，レーザ光を粒子に照射し，散乱光強度の角度分布を解析して粒度分布を求める．解析には種々のアルゴリズムが使われており，同じ試料でもその違いによって測定結果に差がでることがある．

ホリバは「非線形繰返回帰分析法」を採用することによって，この問題を解消した．

the reproducibility of this type of instrument. Both accuracy and reproducibility point to the proper selection of the algorithm.

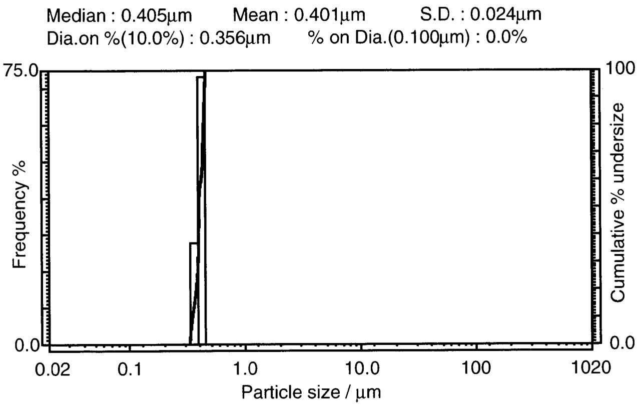


Fig.2 Analysis of 0.405 $\mu$m PSL standard

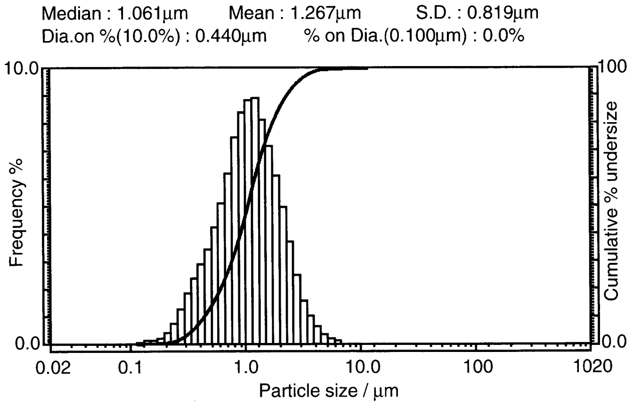
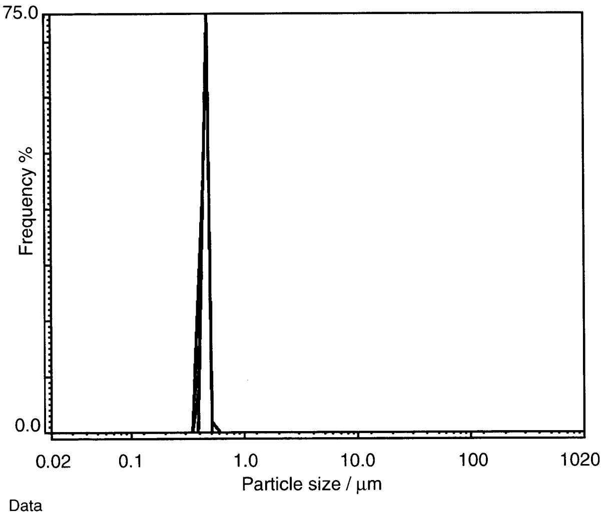
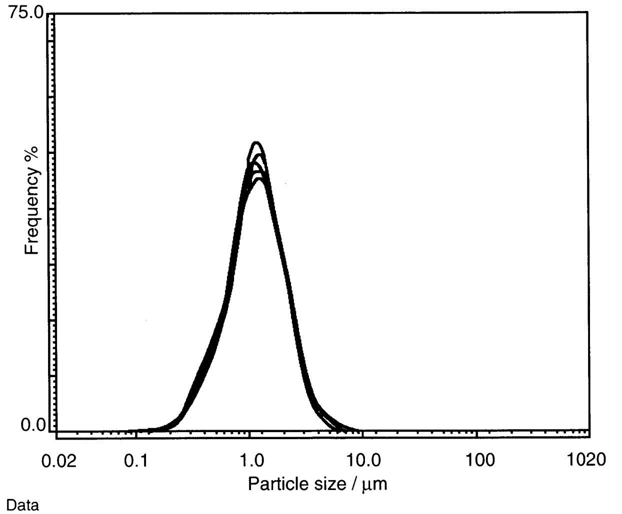


Fig.3 Analysis of BCR-66 quartz standard



| Data Memory | Hist. | Cum. | SAMPLE NAME | ID# | Median(µm) |
|---|---|---|---|---|---|
| Main | —— | | 405 nm NIST-traceable standard | 951013-145 | 0.405 |
| M01 | —— | | 405 nm NIST-traceable standard | 951130-950 | 0.416 |
| M02 | —— | | 405 nm NIST-traceable standard | 950830-003 | 0.411 |
| M03 | —— | | 405 nm NIST-traceable standard | 951201-004 | 0.414 |
| M04 | —— | | 405 nm NIST-traceable standard | 950929-006 | 0.416 |

Fig.4 Multiple runs of 0.405 $\mu$m PSL standard



| Data Memory | Hist. | Cum. | SAMPLE NAME | ID# | Median(µm) |
|---|---|---|---|---|---|
| Main | —— | | BCR 66 standard | 951013-149 | 1.061 |
| M01 | —— | | BCR 66 standard | 951130-958 | 1.061 |
| M02 | —— | | BCR 66 standard | 951114-013 | 1.082 |
| M03 | —— | | BCR 66 standard | 951121-033 | 1.073 |
| M04 | —— | | BCR 66 standard | 951201-012 | 1.070 |

Fig.5 Multiple runs of BCR-66 quartz standard

## 4. 実測例

当社の粒度分布測定装置(LA-910)で石英とポリスチレン標準試料を測定すると，測定値と標準値との間に優れた相関性(図2,3)と，高い再現性(図4,5)が得られており，本アルゴリズムの正しさが証明されている．

火炎法による$SiO_2$原料製造の原理(図6)と製造工程(図7)，および生成物の電子顕微鏡(TEM)像を示す(図8,9)．元の$SiO_2$粒子は小さい(20nm以下)が，火炎の中で凝集し，さらに，溶液中での挙動も考慮しなければならない(図10)．スラリの特性は粒子の凝縮状態(ネットワーク)によって左右され，凝縮度は剪断エネルギーで決まる．

入荷直後のスラリは，平均粒径には差がないが，1.0$\mu$m以上の割合は大きく異なる(図11,12)．ウエハの傷は大粒子が問題と言われているが，実験的には確認されていない．

In order to investigate typical applications of $SiO_2$ slurries, the chemical nature of the $SiO_2$ must be understood. In Figure 6[14] the procedure for preparing the raw $SiO_2$ is shown. The process for its production is shown below in Figure 7[14].

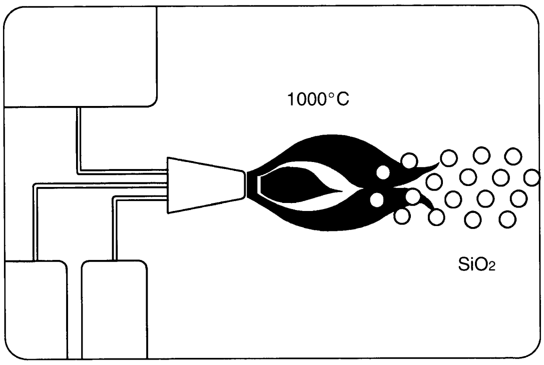


Fig.6 Aerosol flame(Schematic)

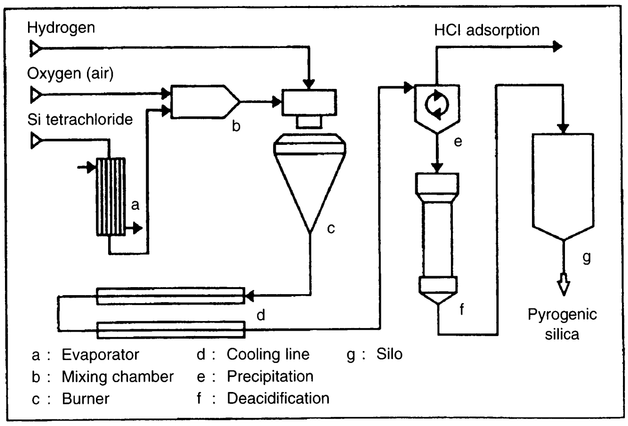


Fig.7 Production of aerosol(Flow chart)

Typical products of this process are shown in Figures 8 and 9. These two photographs reveal the complicated nature of the individual particles of $SiO_2$. The fundamental particles are truly small(~20nm), but they aggregate in the flame. These aggregates are the basic units present in the slurry which must be controlled. The shape of the aggregates make it a very difficult system to study and understand.

To make matters even worse, one must account for the behavior of the aggregates in solution. This added complexity is shown in Figure 10[14].

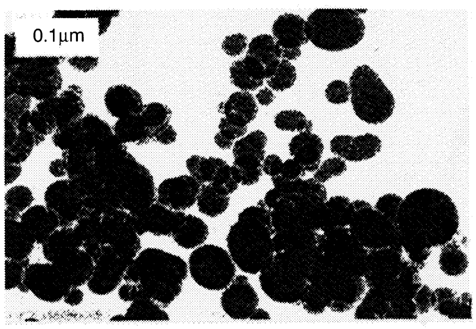


Fig.8 TEM of aerosol 0 x 50

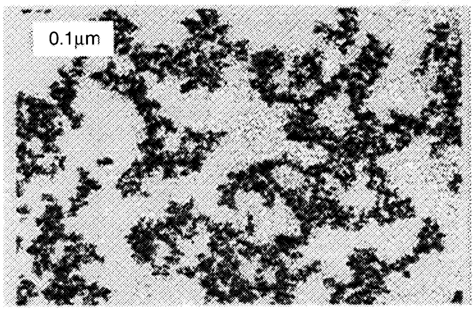


Fig.9 TEM of aerosol 380

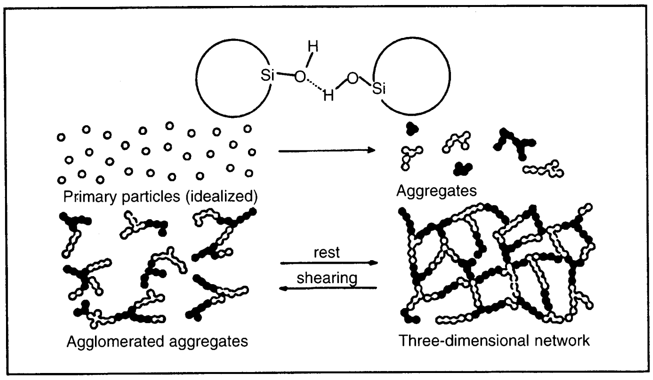


Fig.10 Schematic representation of the interaction between aerosol particles in liquids

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LA-910によるスラリ供給系の特性監視への適用例を示す．不合格のスラリ供給系には1.0μm以上の粒子が多量に含まれており(図13)，供給系を軽く洗浄しても完全には取り除けないが(図14)，徹底的な洗浄によって完全に除去されている(図15)．

## 5. まとめ

スラリの特性解析には，粒径分布を単独に求めるだけでは不十分である．粒径と性能との関係，例えば，エッチング速度，残留スラリ量，傷の数などを求める必要がある．これには，メーカのノーハウが絡んでおり今後の課題である．

(抄訳　編集部)

The figure clearly shows that the nature of the aggregates depends upon the energy state of the system containing the slurry. High shear energy retains the agglomerated particles as they were produced. Low energy allows the aggregates to agglomerate even further. Thus the performance would be characterized by this so called "network" of particles. This behavior makes performance prediction a very difficult procedure.

The investigation of CMP slurries must begin with the routine slurries which are being produced and used on an everyday basis. Figures 11 and 12 show some typical as received slurry samples.

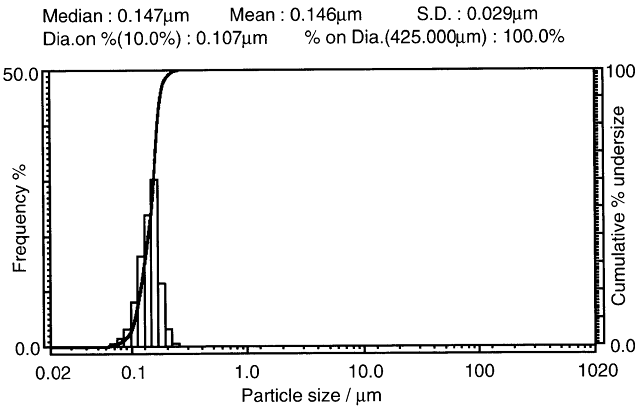

Fig.11 Fumed silica A sample

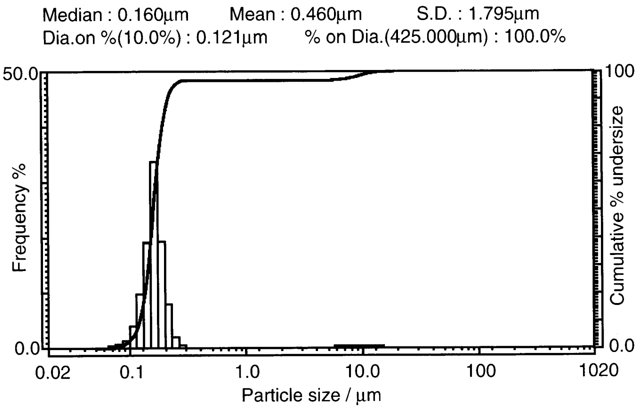

Fig.12 Fumed silica B sample

While the median size does not show any significant difference, the volume percentage of particles greater than 1.0 $\mu$m is drastically different. Since scratching by large particles is a major source of concern, these two slurries should behave quite differently. The experiments to validate this hypothesis remain to be performed.

Since the presence or absence of large particles is so clearly detected by this instrument, other applications were examined. A good example is presented in Figures 13,14 and 15.

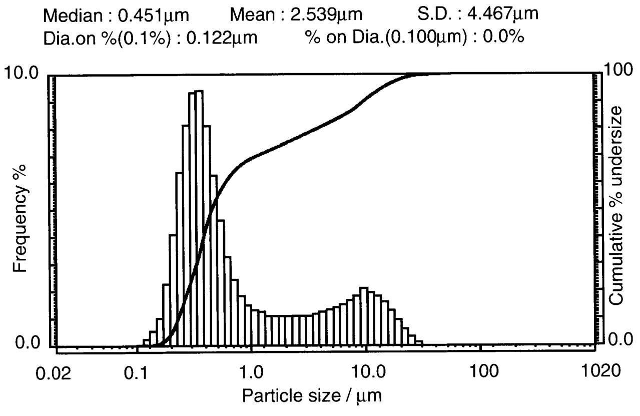

Fig.13 Slurry prior to flushing

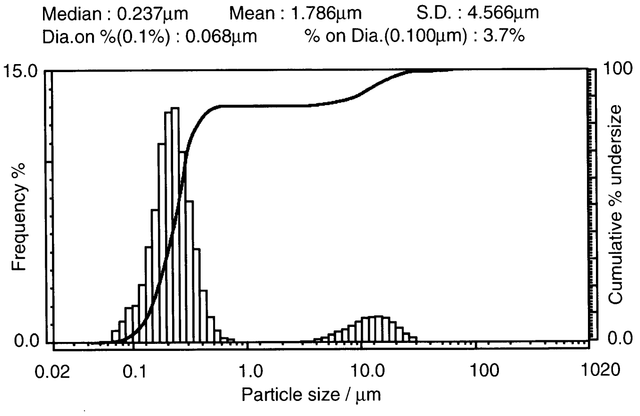

Fig.14 Slurry after one flushing procedure

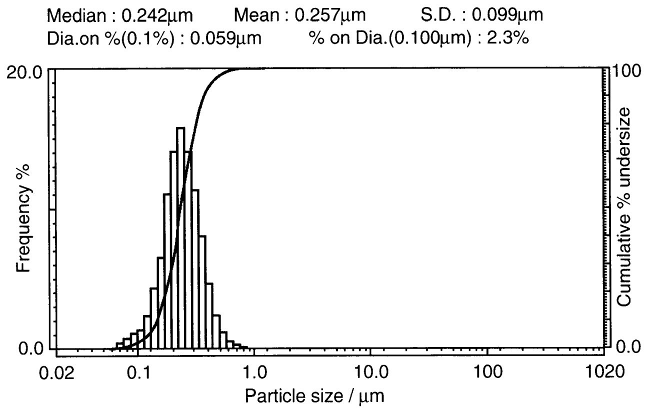

Fig.15 Slurry after multiple flushing procedures

This represents a procedure for monitoring the removal of a poorly performing slurry from a CMP delivery system. In Figure 13 the particle size of an unacceptable slurry is presented. Note the large quantity of particles greater than 1.0 $\mu$m. This slurry was removed and the system was cleaned by a standard procedure. In Figures 14 good slurry was added, but analysis revealed that particles greater than 1.0 $\mu$m remained. The entire system was thoroughly cleaned and additional slurry was added.

Figure 15 shows that all of the large particles were now removed. The system was now ready for further use.

## 5. Summary

These examples show the ability of particle size distribution analyzers to analyze slurry samples. Unfortunately, particle size as a physical test only provides half of the required information. As with any physical test, the most critical part is correlating the size to a performance criteria[15]. In the case of CMP slurries it could be etch rate, residual slurry trapped, number of micro-scratches, etc. This is truly where the arduous work begins. One must prepare a large number of slurries and then measure the performance characteristics with them. The final result is a powerful predictive tool to specify performance. Presumably many such tests have been performed, but the proprietary nature of the work has prevented it from being published.

<Reference>

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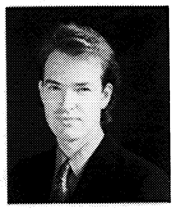

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