Methodology for reducing process variability through in-situ production of positive photoresist developer

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ABSTRACT

Process variability in a photolithographic process can arise from a number of sources including photoresist developer assay variation. A change in activity of only ± 0.001 eq/l in a developer solution with a normality of 0.2624 eq/l can consume or exceed the allowable ± 5.0% CD specification set by most fabs. This change, which represents a relative error (error/setpoint) at 3σ of only ± 0.4%, is typical of the allowable variability from developer suppliers.

Developer production systems with tighter assay tolerances should reduce process variability and increase process robustness. This study compared the effectiveness of several control techniques for blending tetramethyl ammonium hydroxide (TMAH) developer. Techniques with feedforward and feedback control were investigated.

An analysis of sources of error in developer blending systems indicated that feedforward techniques could not achieve the desired relative error of ± 0.4%. An experimental study was undertaken to determine the relative error of three feedback control methods. The feedback techniques yielded considerably tighter assay control than that expected from the feedforward techniques. An analog setpoint control algorithm used with conductivity measurements provided more precise control than a discrete setpoint control algorithm. However, only feedback control with titration met the goal, achieving a relative error of only ± 0.13% at 3σ (± 0.00034 eq/l).

Keywords: positive photoresist developer, tetramethyl ammonium hyrdoxide, assay control, on-site chemical blending, titration, conductivity

2. INTRODUCTION

A well controlled photolithography process is essential in semiconductor fabrication. As circuit geometries have become smaller and circuit patterns have become more complex, tolerances have shrunk dramatically. Most fabs have set a ± 5.0% critical dimension (CD) specification. This has driven the search for improved control of all parts of the photolithography process, including consistency in developer assay.

TMAH is frequently used as a positive photoresist developer. A change in activity of only ± 0.001 eq/l in a TMAH solution with a normality of 0.2624 eq/l (2.38% by weight) can exceed the allowable ± 5.0%

CD specification. This change in concentration represents a relative error (error/setpoint) of only ± 0.4% at 3σ. To help meet the rigorous CD specifications, the assay of TMAH-based developer solutions should be within this limit.

Many factors can affect the normality of positive photoresist developers such as TMAH. Manufacturing variables such as sampling techniques, operator variability, tank residence time and batch homogeneity can all influence the final concentration of developer, even after it has passed final quality control. Packaging materials, packaging integrity, transportation and storage can cause further variability. Additionally, variation can arise within the fab from absorption and chemical extraction.

Many of these sources of variation can be eliminated by on-site blending of developer solutions. All problems associated with packaging, shipping and long term storage are also avoided. In addition, on-site blending gives fab management the opportunity to monitor and control assay variation of their photoresist developer. Blending processes with feedback control allow production of a developer with assay that is independent of variation in incoming chemical assay. In addition, the cost of the TMAH developer prepared on-site is reduced because it is no longer necessary to purchase, ship and store large quantities of a dilute solution.

3. ANALYSIS OF BLENDING APPROACHES

Most methods of on-site blending currently used share the advantages of improved purity and reduced cost of blended chemical. On-site chemical blending uses ultrapure deionized water and concentrated chemical to blend to the desired concentration of chemical. However, there are several different approaches used in on-site blending to control blend assay. Feedforward blending methods use process variables that are subject to the inherent error in the incoming chemical assay, since these methods do not measure the assay of the blend directly, e.g. blending by weight or volume. Other blending methods use process variables that measure the assay of the blend and thus are independent of variations in incoming chemical assay. These blending methods utilize feedback control with respect to TMAH assay, e.g. blending by conductivity or titration.

Measurement of TMAH concentration in blending systems can be accomplished using either conductivity or titration. Titration has the advantage of being directly referenced to a primary concentration standard. Unfortunately, titration measurements require significant analysis time. Blending systems which use conductivity for end point determination require minimal analysis time, but must rely on titration for calibration of probes and meters. Additionally, conductivity does not measure concentration as accurately as titration for several reasons, including sensitivity to temperature variation.

Error analysis in this study was based on the orthogonal addition of errors[1]. This method is used to combine normally distributed errors from independent sources. The total error in feedforward blending systems is a combination of the errors attributed to the capability of the blending system and incoming TMAH assay variation, as determined by the following equation:

$$E_{total} = (E_{system}^{2} + E_{assay}^{2})^{1/2} \quad (1)$$

The results of this study are presented as relative error, that is, as the ratio of total error to set point, expressed as a per cent.

The assay error in feedforward systems based on weight or volume measurement was calculated to be ± 1.5-2.5%. Variation in the incoming chemical concentration was assumed to be ± 1-2%. The system error was estimated at ± 1.0-1.5%. Even under optimum conditions, feedforward techniques based on weight or volume of blend components cannot achieve the desired accuracy of ± 0.4%.

4. EXPERIMENTAL EVALUATION

An experimental study was undertaken to determine the relative error of developer blending systems with feedback control. Blending systems with titration and conductivity were included. In addition, two methods of achieving conductivity end points were used. Discrete setpoint control relies on independent hardware with relays to determine when the blend is within the setpoint range. Use of an analog PLC-based control provides improved resolution of conductivity signal, filtering and conditioning of the raw data. It was expected that use of analog setpoint control would result in improved accuracy.

TMAH (25% by weight) and DI water were blended to produce 0.2624 eq/l TMAH (2.38% by weight). TMAH was blended in 25 gallon batches in the patent pending FSI International ChemBlend 100™. Feedback systems based on conductivity with discrete setpoint control used a proprietary control system; feedback with analog setpoint control used a PLC-based control system. In tests using titration for feedback control the ChemBlend system was equipped with an integrated on-line autotitrator with analog feedback. Thirty batches were prepared with each control approach to ensure that the data were normally distributed as predicted by the central limit theorem. In all cases, TMAH assay of the completed blend was determined in triplicate using a separate titrator (Metrohm 716 DMS Titrino). Titration was performed with a single lot of titrant which was referred back to a primary standard.

5. RESULTS

The effect of feedback control method on relative error of the TMAH blend is presented in Figures 1 through 3, which present the concentration variation in TMAH blends prepared using different control methods. In all cases the setpoint was 2.38% by weight. Upper and lower design control limits (UCL and LCL), ± 0.4% (± 0.001 eq/l), are shown for reference. In addition, each process's 3σ control limits are shown.

Figure 1 shows the concentration of blends prepared using conductivity with discrete setpoint control. The relative error at 3σ was ± 0.76% (± 0.0020 eq/l). The 3σ control limits were well outside the design control limit.

Figure 2 presents data obtained using conductivity with analog setpoint control. The change to analog control resulted in an improvement in process repeatability. The relative error of this method at 3σ was ± 0.50% (± 0.0013 eq/l), representing a 0.26% decrease in relative error over the use of discrete setpoint control. However, it did not reach the goal of ± 0.4% (3σ).

Figure 3 presents data obtained using titration feedback control. This method resulted in a significant improvement in process repeatability. The relative error at 3σ of this control system was ± 0.13% (± 0.00034 eq/l) at 3σ, well within the process design control limits of ± 0.4% (3σ).

6. DISCUSSION

A summary of the results is presented in Table I. The relative error calculated for feedforward control methods has been included for comparison. Although feedback control based on conductivity is superior to feedforward techniques, it does not meet the target relative error of ± 0.4% (± 0.001 eq/l). Only feedback control based on titration provided the required repeatability. With a relative error of only ± 0.13%, feedback control based on titration is well within the specification. A relative error of ± 0.13% is equivalent to an assay error of only ± 0.00034 eq/l at 3σ.

The four control methods can also be evaluated by statistical process control analysis. A process's capability (C_P) is the ratio of the specification tolerance to the range containing three standard deviations, as follows:

$$C_P = \frac{\text{Specification tolerance}}{3\sigma} \quad (2)$$

Systems with a $C_P \leq 1$ are not statistically in control. Some companies have established minimum capability requirements of 1.33 or greater[2]. Motorola's 6σ capability goal requires a C_P of ≥ 2.0.

Table II contains the C_P of each blending control method determined from the experimental data. Only feedback control using titration provides the required process capability. With a C_P of 3.08, a blending system with feedback control based on titration will be able to meet the more stringent tolerances that are expected in the future.

7. CONCLUSION

Four methods of blending system control were evaluated for their ability to provide precise TMAH developer blends. An assessment of photolithography requirements indicated that blending system control methods must provide ± 0.4% (± 0.001 eq/l) relative error in a 2.38% TMAH blend. Calculation of the relative error associated with feedforward control techniques showed that they could not provide the desired relative error. The error associated with feedback control techniques was determined experimentally. An analog setpoint control algorithm provided more precise control than a discrete setpoint control algorithm, resulting in a decrease in relative error from ± 0.76% (± 0.0020 eq/l) to ± 0.50% (± 0.0013 eq/l). Neither conductivity control technique could provide the required precision. Only feedback control based on titration reached the requirements of the photolithography industry. The relative error of a blending system using feedback control with titration had a relative error of ± 0.13%, which is equivalent to an error of only ± 0.00034 eq/l at 3σ. An analysis based on statistical process control revealed that the C_P of blending using feedback with titration control is 3.08. This technique has the capability to meet current lithography requirements, and to accommodate stricter requirements expected in the future.

8. REFERENCES

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2. Process Control, Capability and Improvement, IBM Corporation, New York, 1984.

Table I. Relative and absolute error associated with control techniques used in TMAH developer blending

Technique	Relative Error at 3σ	Assay Error at 3σ (eq/l)
Feedforward (calculated)	1.5-2.5%	0.0039-0.0066
Feedforward (without incoming chemical assay variation)	1.0-1.5%	0.0026-0.0039
Feedback using conductivity with discrete setpoint control	0.76%	0.0020
Feedback using conductivity with analog setpoint control	0.50%	0.0013
Feedback using titration	0.13%	0.00034

Table II. Process Capability with different control techniques used in TMAH developer blending

Technique	C_P
Feedforward (calculated)	0.16-0.27
Feedforward (without incoming chemical assay variation)	0.27-0.40
Feedback using conductivity with discrete setpoint control	0.53
Feedback using conductivity with analog setpoint control	0.80
Feedback using titration	3.08

Figure 1: TMAH Concentration Variability Using Conductivity Feedback Control (Discrete)

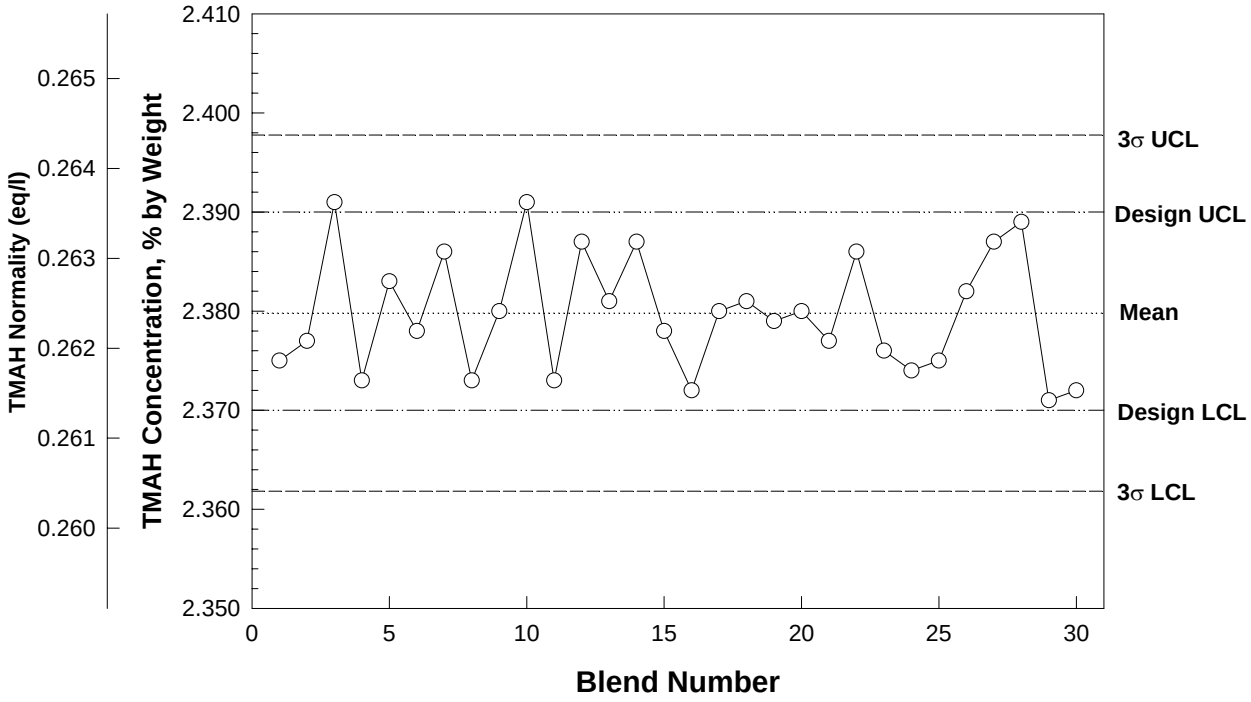

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Figure 2: TMAH Concentration Variability Using Conductivity Feedback Control (Analog)

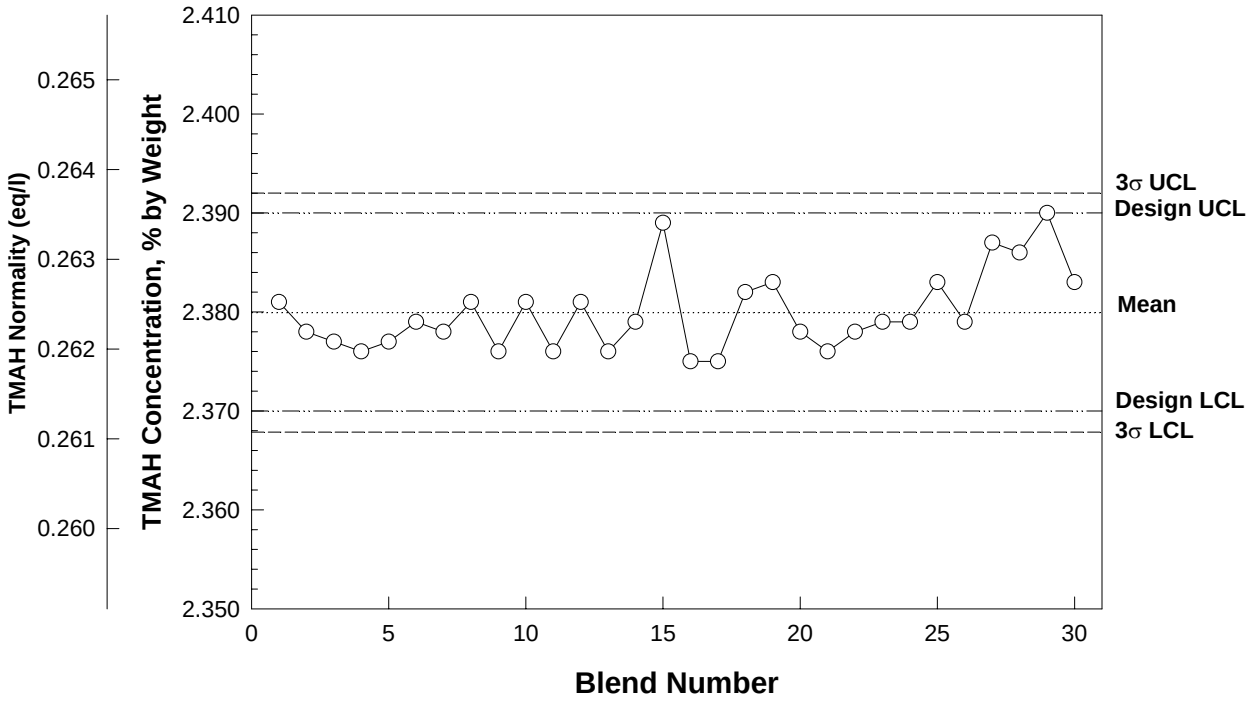

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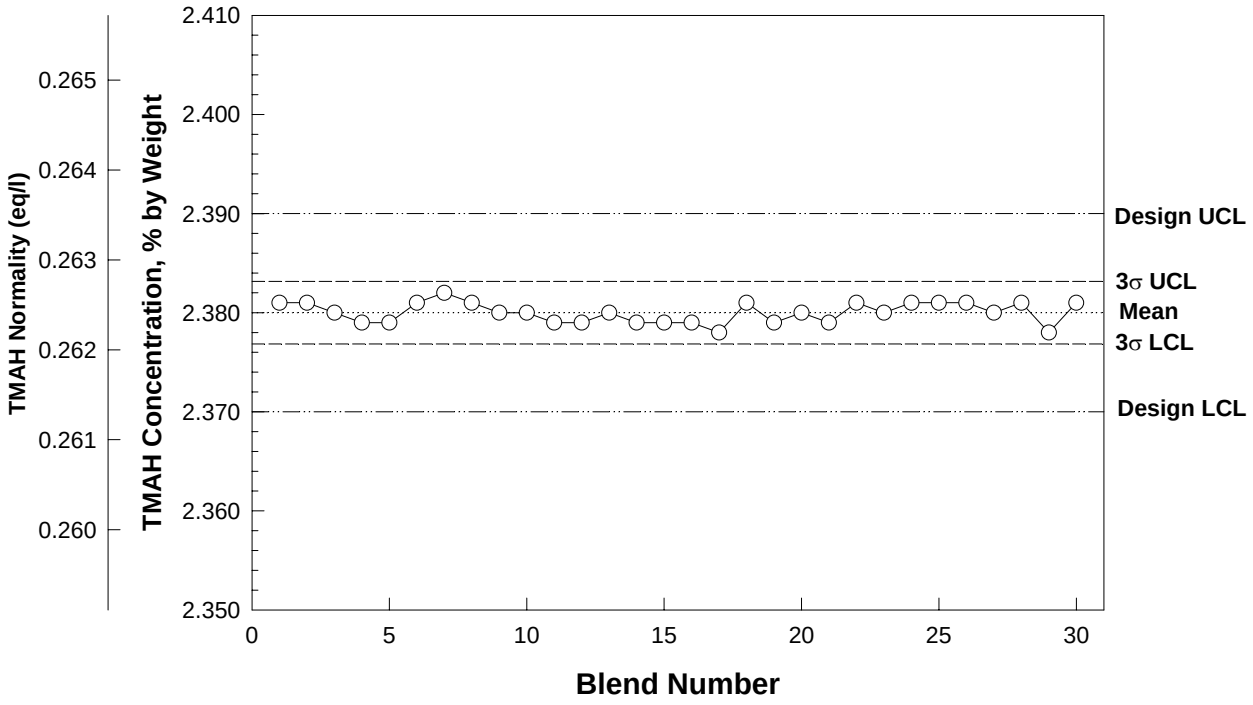
Figure 3: TMAH Concentration Variability Using Titration Feedback Control



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