DOWEX™ Ion Exchange Resins

UPCORE™ System Reduces Boron to Undetectable Levels in Ultrapure Water

Site Information

Location:
Dresden, Germany

Purpose:
Reduce boron levels to meet ultrapure water specifications

Start-up:
1998

Performance:
Boron levels reduced to below detectable, enabling plant to meet critical specifications for process water

The Fab 30 plant in Dresden, Germany, uses the UPCORE™ system in its demineralizer unit to reduce ions, silica, boron, and TOC levels in process water. (Photo courtesy of AMD)

Introduction

The Fab 30 plant in Dresden, Germany, is AMD's main microprocessor facility and the largest microprocessor plant in Europe. Although the production of microprocessors has always placed stringent quality demands on process water, the move to submicron devices adds additional challenges. Research has shown that boron in process water can cause poor P-N junction and can reduce the production yield of submicron devices.[1] Unfortunately, boron is difficult to remove with conventional water-treatment systems, and boron compounds can have negative effects on the water-treatment system itself.[2] A specially designed system that focuses on boron removal requirements enabled Fab 30 to meet its ultrapure water (UPW) specifications.[3]

Background

A conventional UPW treatment plant purifies water with a two-pass reverse osmosis (RO) system followed by a two-pass mixed-bed ion exchange (IX) system. Typically, silica breakthrough signals exhaustion of the IX beds and prompts regeneration. However, a study at AMD's Submicron Development Center showed that boron breaks through earlier than silica,[2,3] making boron the most critical parameter for determining exhaustion of IX beds in a boron-sensitive UPW system.

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Background, cont.

Removal efficiency of boron depends on pH and the concentration of boron in the solution. Boric acid is a weak acid which is nearly non-dissociated at pH <7 and strongly dissociated at pH >11.5. The more strongly that boron is dissociated, the more easily it can be removed. However, improving boron rejection by raising the pH of the feed water to the RO system with NaOH results in decreased sodium rejection and an increased sodium load to the mixed beds. The only alternative in a conventional system is to run the plant at a pH that balances enhanced boron removal against increased sodium levels.

Fab 30 UPW Plant Design

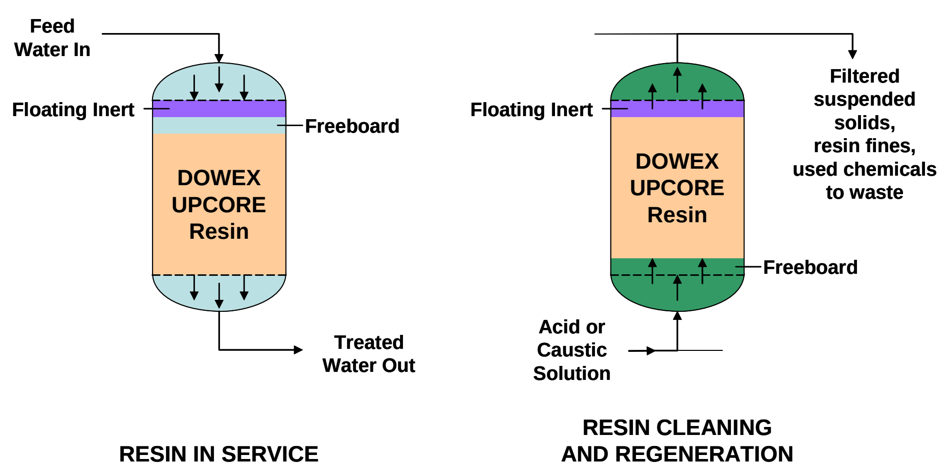
The design of the Fab 30 UPW system focused on boron removal. Instead of the conventional RO→IX mixed-bed design, the UPCORE™ system was installed as a first-pass treatment. The UPCORE system is based on the following principles:

- Counter-current ion exchange technology
- Packed bed design
- Upflow regeneration/downflow service
- Uniform particle size (UPS) resin technology

UPCORE™ System

In the service cycle, a wide operational flow flexibility is possible. In this cycle, the feed water enters the vessel from the top (Figure 1). Before regeneration, compaction water flows at high velocity from the bottom to the top and compacts the resin bed against the inert resin and upper nozzle plate. Without flow interruption, the regenerant and subsequently the rinse water passes through the resin bed in an upflow direction. There is no need for a separate backwash tank because the suspended solids are automatically removed from the surface of the resin bed during the compaction step of each regeneration cycle.

Figure 1. Diagram of the UPCORE system



The advantages to the UPCORE system include

- Excellent water quality
- High chemical efficiency
- Short regeneration time
- Simple construction and control
- Self cleaning
- Insensitivity to production flow variations and stops
- No risk of carry-over of resin fines
- Anion resin layered beds without the need for a middle plate

UPCORE™ System, cont.

All ion exchange vessels are equipped with sample ports for boron analysis, which is measured by inductively coupled plasma-mass spectrometry (ICP-MS). When the plant was installed in 1997, the detection limit for boron was 50 ppt.

Reverse Osmosis and Mixed Beds

In the Fab 30 UPW plant, the UPCORE™ system is followed by an RO system and primary and polishing mixed-bed IX systems. The RO system includes equipment to enhance the pH in the RO feed to achieve a higher boron rejection.

The RO unit uses FILMTEC™ SG30LE-430 semiconductor grade elements.

The primary mixed beds use DOWEX™ UPW resins. (Photos courtesy of AMD)

Resins and Elements

Table 1 lists the resins and elements used.

Table 1. Resins and elements used in Fab 30 UPW treatment system

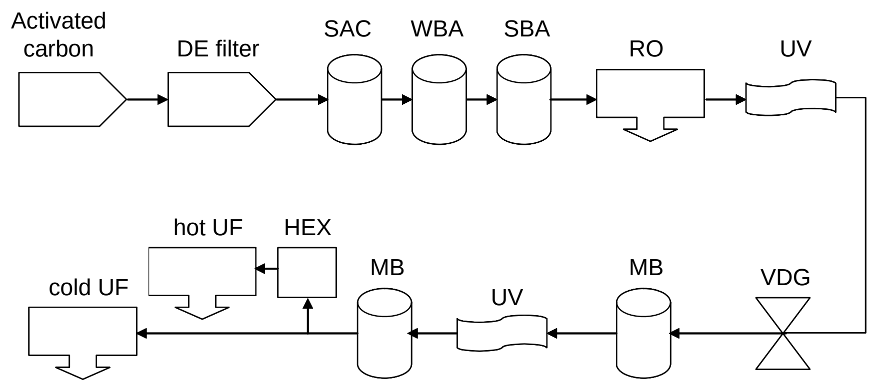
Treatment System	Resin/Membrane	Configuration/Flow
UPCORE		
Strong acid cation resin	DOWEX UPCORE Mono C-600	4 lines at 82.5 m^3/h (363 gpm)
Weak base anion resin	DOWEX UPCORE Mono WB-500	
Strong base anion resin	DOWEX UPCORE Mono A-625	
Reverse osmosis	FILMTEC SG 30 LE-430 UPW	11 lines at 35 m^3/h (154 gpm)
Mixed bed		
Primary	DOWEX MONOSPHERE™ 650C UPW (H) and DOWEX MONOSPHERE 550A UPW (OH)	4 x 100 m^3/h (441 gpm)
Polishing	DOWEX MONOSPHERE 650C UPW (H) and DOWEX MONOSPHERE 550A UPW (OH)	2 x 165 m^3/h (364 gpm) with two mixed beds in series

Objectives of these design parameters were:

- Improve boron rejection at an early stage
- Enhance pH upstream of the RO to optimize boron rejection by RO system
- Provide sample ports for early detection to prevent any ionic breakthrough at the ion exchange system
- Achieve a high-quality RO reject that could be easily used in nonprocess applications, such as make-up water for cooling towers and air scrubbers

Process Steps

Figure 2. Process steps for Fab 30 UPW plant



DE = diatomaceous earth
SAC = strong acid cation resin
WBA = weak base anion resin
SBA = strong base anion resin
RO = reverse osmosis

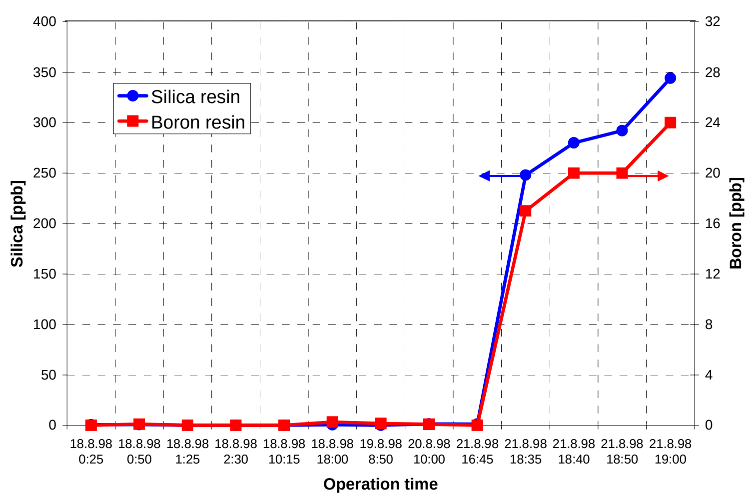
UV = ultraviolet sterilizer
VDG = vacuum degasifier
MB = mixed-bed ion exchange
HEX = heat exchanger
UF = ultrafiltration

Plant Operation

The raw water used in Fab 30 can change significantly depending on the primary water source feeding the UPW plant's raw water tank. The conductivity of the raw water can fluctuate in the range of 350–800 µS/cm, boron levels in the range of 20–180 ppb, and SiO_2 as high as 8–15 ppm. TOC levels range from 10–15 mg $KMnO_4$/L. After filtration through activated carbon filters and precoat filters, the water is fed to the make-up IX trains. The IX trains upstream of the RO units were intended to be regenerated after SiO_2 breakthrough detection at the sample ports. A safety regeneration occurs if the conductivity of the sample probe increases. After stabilizing the operation of the plant in the commissioning phase (May to August 1998), first breakthrough characteristics with regard to boron and silica were evaluated.

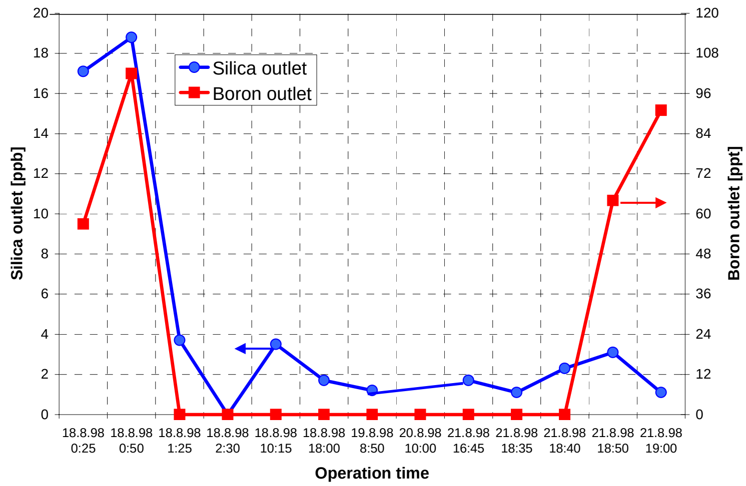
Figures 3 and 4 show the relationship of boron to silica breakthrough across single-bed IX units. The x-axis (operation time) of the diagrams is not linear.

Figure 3. Boron and silica breakthrough at resin train 2 of cation/anion exchanger



Plant Operation, cont.

Figure 4. Boron and silica breakthrough at outlet train 2 cation/anion exchanger



The data collected during start-up in 1998 under normal operating conditions showed that almost no boron (50–100 ppt) was detected in the effluent of the single-bed IX train. Later data showed a stable boron level of <30 ppt in the outlet of the IX train. The first two data sets show the fast rinse phase conditions prior to the train being set into operation.

The SiO_2 breakthrough approaching the sample ports showed a significant increase of ionic silica and an increasing portion of colloidal silica and boron concentration reaching this part of the resin bed in parallel. When the silica front reached the probe, no boron breakthrough through the exchanger (outlet) was observed. Approximately 30 min later, however, the boron level had risen to approximately 0.1 ppb in the outlet. After that point, regeneration was initiated and the test terminated.

Regeneration occurs based upon a fixed throughput through the trains. Boron data since January 1999 have not shown any detectable boron in the outlet of the IX trains, the RO permeate, or the primary outlet of the polishing mixed beds.

Conclusions

AMD's Fab 30 plant demonstrates that with the right design, operating parameters, and use of Dow's counter-current UPCORE™ technology, it is possible to reduce boron below the detection limit (2003: < 20 ppt) at an early make-up stage of the water treatment plant. After more than 5 years continuous operation, AMD Fab 30 has not observed any detectable boron in the outlet of the IX demineralization unit.

Further steps implemented to enhance boron removal efficiency, such as increasing the pH of the water before it reached the RO unit, were eliminated when boron removal after demineralization was stabilized. Elimination of caustic addition upstream of the RO unit and the subsequent increased cycle length of the regenerable mixed beds (up to several months) led to significant savings of chemicals and service water.

Based on the excellent performance at Fab 30, AMD is considering installing the same boron removal technology for the new Fab 36, which will start up during 2004/2005 at the same site.

References

1. Yagi, Y.; Hayashi, F.; Uchitomi, Y.; “Evaluation of boron behavior in ultrapure water manufacturing system,” Semiconductor Pure Water and Chemical Conference; San Jose; California (March 8-10, 1994)
2. Malhotra, S.; Chan, O.; Chu, T.; Focsko, A.; “Correlation of boron breakthrough versus resistivity an dissolved silica RO/DI system,” Ultra Pure Water 13, pp.22-26 (May/June 1996).
3. Sauer, R.; Gensbittel, D.; Bartz, R.; Rompf, F.; Ziemer-Popp, Ch.; Wilcox, D.; Malholtra, S.; “Boron removal experiences at AMD,” Ultra Pure Water, pp. 62-68 (May/June 2000)

DOWEX™ Ion Exchange Resins
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