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(DE96011355)

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Topical Report
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Work Performed Under Contract No.: DE-AC21-88MC25034

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Westinghouse Science And Technology Center
1310 Beulah Rd.
Pittsburgh, PA 15325

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M. A. Alvin

July 25, 1995

ABSTRACT

Advancements have been made during the past five years to not only increase the strength of the as-manufactured clay bonded silicon carbide candle filter materials, but also to improve their high temperature creep resistance properties. This report reviews these developments, and describes the results of preliminary qualification testing which has been conducted at Westinghouse prior to utilizing the advanced clay bonded silicon carbide filters in high temperature, pressurized, coal-fired combustion and/or gasification applications.

INTRODUCTION

Field Experience

High temperature particulate removal systems have been designed and operated by Westinghouse for use in advanced coal-fired process applications. Typically monolithic, first generation, clay bonded silicon carbide Pall Vitropore 442T candles were tested for a maximum of 1341 hours in the Ahlstrom pressurized circulating fluidized-bed combustion (PCFBC) pilot plant in Karhula, Finland.(1) At PCFBC operating temperatures of 830°C (1525°F), the Pall Vitropore 442T candles experienced high temperature creep (i.e., maximum elongation of 26 mm), and subsequently crack growth at the base of the filter's flange, and ultimately catastrophic failure.

During operation at the American Electric Power (AEP) Demonstration Plant, eight Pall Vitropore 442T filter elements were installed and operated in the 660-760°C (1220-1400°F) W-APF system during conduct of Test Segment #4 (July 1994-October 1994). Three of the Pall Vitropore 442T filter elements were reinstalled in the W-APF, and were successfully operated for a period of 1110 hours at temperatures of 760-845°C (1400-1555°F) in Test Segment #5 (January 1995-March 1995).(2) Post-test inspection indicated that the Pall Vitropore 442T filters elongated by 17-20 mm after 2815 hours of operation at AEP. Cracks were not evident along the elongated Pall Vitropore 442T candle filter body (i.e., particularly below the flange).

During operation in Test Segment #5, 150 newly manufactured Pall Vitropore 442T candle filter elements were also installed in the W-APF system. All candles remained intact after 1110 hours of operation. Post-test characterization of seventeen elements indicated that the filters elongated by 0-4 mm. Since the filter elements were identically manufactured for use in both test segments at AEP, the limited elongation of the clay bonded silicon carbide filter matrix after 1110 hours of operation in the 760-845°C (1400-1555°F), PFBC environment in test Segment #5 vs 17-20 mm elongation after 1705 hours of operation in the 660-760°C (1220-1400°F) PFBC environment implies that a lag time is required to initiate high temperature creep in the binder containing Pall Vitropore 442T filter matrix.

Throughout the entire test program effort at AEP, Westinghouse included Schumacher Dia Schumalith F40 candles within various filter arrays. As surveillance filters, the Schumacher Dia Schumalith F40 candles successfully achieved 5855 hours of operation in the 620-845 ° C (1150-1555 ° F) PFBC environment between October 1992 and March 1995.

In order to ascertain whether the Schumacher Dia Schumalith F40 filter matrix experienced creep during operation in the PFBC environment, the overall length of the surveillance filter elements were measured intermittently during outages, and compared to their initial as-manufactured lengths. After 5855 hours of operation in the W-APF at AEP, the 1.5 m elements were observed to have elongated by 5-7 mm.[a] Cracks were not evident along the external surface of the filter elements (i.e., particularly below the flange).

Several manufacturing changes have been implemented by the candle filter suppliers as a response to the high temperature creep and possible strength issues encountered during field operation. Both Schumacher and Pall reformulated their binder composition and/or changed the grit size in their clay bonded silicon carbide filter matrices, producing not only stronger, but also higher temperature, creep resistant matrices for potential use in >800 ° C (>1475 ° F) coal-fired process applications. To date, the improved Schumacher Dia Schumalith FT20 filters have been successfully operated for 1705 hours in the W-APF system at AEP at temperatures of 660-760 ° C (1220-1440 ° F; Test Segment #4). Currently field test information is not available for the more recently developed Pall 326 candle filters.

Based on recent materials evaluation conducted at Westinghouse, as well as at the various filter supplier test facilities, and preliminary HTHP testing at Westinghouse, both the Schumacher Dia Schumalith FT20 and Pall 326 filter elements are expected to demonstrate improved operating performance and extended life at 900 ° C (1650 ° F) in the PCFBC test facility in Karhula, Finland.

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[a] *Elongation of 1 mm after 3038 hours of PFBC operation; Elongation of ~3 mm after 4734 hours of PFBC operation.*

Materials Development

Schumacher traditionally manufactured the Dia Schumalith F40 clay bonded silicon carbide candle filter matrix. The candle consisted of an aluminosilicate fibrous outer membrane which covered coarse, underlying support, silicon carbide support grains. A densified transition section of finer silicon carbide grit was incorporated into the body as part of the filter flange construction (i.e., 73-125 mm; ~3-5 inches) in order to provide added strength to the filter matrix. The wall thickness of the Schumacher Dia Schumalith F40 filter was 15 mm.

As the need arose to produce a stronger filter body with improved high temperature creep resistance properties, Schumacher modified the composition of the clay binder, and reduced the silicon carbide grit size within the dimensionally changed 10 mm support wall. The traditional aluminosilicate fibrous membrane was applied to the outer surface of the filter element. In the Schumacher FT20 filter, the densified flange transition section was eliminated.

In order to provide a smoother, non-fibrous outer membrane surface to the Schumacher product line, an oxide-based, granular membrane was developed and applied to the finer silicon carbide grit, 10 mm support structure, producing what was designated as the Schumacher 10/20 filter matrix. The binder in the Schumacher 10/20 filter was comparable to that used to manufacture the Schumacher Dia Schumalith F40 candles. As a by-product of the advanced filter materials development effort, the improved, high temperature creep resistant binder was incorporated into the 10 mm finer grained silicon carbide support matrix, and the oxide-based membrane was applied to the outer surface of the filter element. The resulting product was designated as Schumacher T10/20. Both the Schumacher 10/20 and T10/20 filter elements were constructed without densified flange transition sections. Product information which is currently available for the Schumacher clay bonded silicon carbide filter materials is provided in Appendix A.

Similarly during the course of development, the clay bonded silicon carbide candle filters that were developed within the U.S. underwent several production changes. Historically, these materials were manufactured by Refractron in Rochester, NY, and were designated as 501 and 505 candle filters. In the late 1980's, further development lead to the production of the Pall Vitropore 442T filter matrix which consisted of a 10 mm coarse support silicon carbide wall that was coated with a finer grained silicon carbide membrane. Appendix B provides an overview of the morphology and composition of the Refractron and early Pall Vitropore 442T filter products.

Recently additional changes have been made to the Pall Vitropore 442T filter to provide improved, high temperature creep resistance properties to the matrix. The higher temperature, creep resistant Pall candle filter is currently designated as 326.

In the following sections, a comparison of the morphology and semi-quantitative elemental composition of the binder phase in the Schumacher and Pall candle filter materials is presented. Similarly, the high temperature creep resistance of the Schumacher and Pall filter materials is reviewed. Preliminary high temperature, high pressure qualification testing which is generally conducted at the Westinghouse Science and Technology test facilities in Pittsburgh, PA, prior to installing and utilizing advanced filters in the field is also described.

MATERIALS CHARACTERIZATION

Filter Matrix Morphology

The morphology of the clay bonded silicon carbide Schumacher Dia Schumalith F40 and the improved, high temperature, creep resistant Schumacher Dia Schumalith FT20 filter matrices are shown in the scanning electron micrographs (SEM) presented in Figures 1 and 2. Both filter elements were manufactured with an aluminosilicate fibrous outer membrane which was sintered onto the underlying support silicon carbide grains. The smaller silicon carbide grains which were utilized to manufacture the Schumacher Dia Schumalith FT20 candle provided additional strength to the filter matrix.

In both the Schumacher Dia Schumalith F40 and FT20 filter materials, a binder phase encapsulated the silicon grains, and formed the bond posts or ligaments between adjacent grains. In the as-manufactured state, the binder in both materials appeared to be smooth and "velvety", lacking crystalline features.

The morphology of the clay bonded silicon carbide filter materials that were manufactured by Pall is shown in Figures 3 and 4. These include the Pall Vitropore 442T, as well as the improved, high temperature, creep resistant material which is currently designated as Pall 326.

Typically the Pall candles were manufactured with a fine grain silicon carbide membrane along the outer surface of the filter elements. Below the membrane were larger silicon carbide grains which formed the support matrix of both materials. Once again, a binder phase encapsulated the membrane and/or support wall silicon carbide grains, and formed the bond posts or ligaments that held adjacent grains to each other.

Generally the morphology of the binder phase was smooth and "velvety" in the as-manufactured Pall Vitropore 442T filter matrix. Limited crystallization was, however, evident in the as-manufactured Pall 326 filter matrix (Figures 4b, 4e, and 4f).

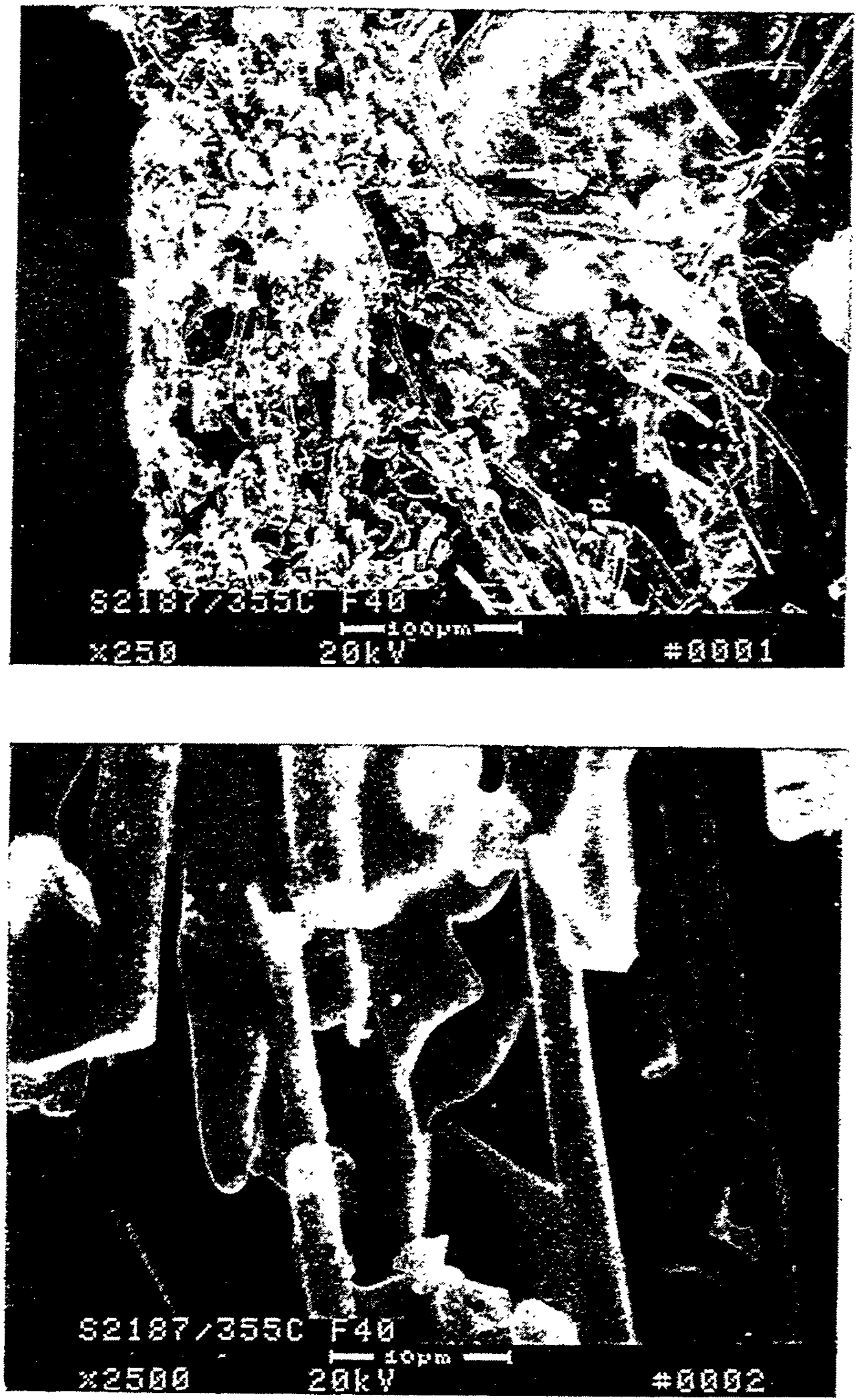


Figure 1a – Morphology Of The Cross-Sectioned Schumacher Dia Schumalith F40 Filter Matrix Illustrating The Affinity Of The Aluminosilicate Fibrous Outer Membrane To The Underlying Support Silicon Carbide Grains

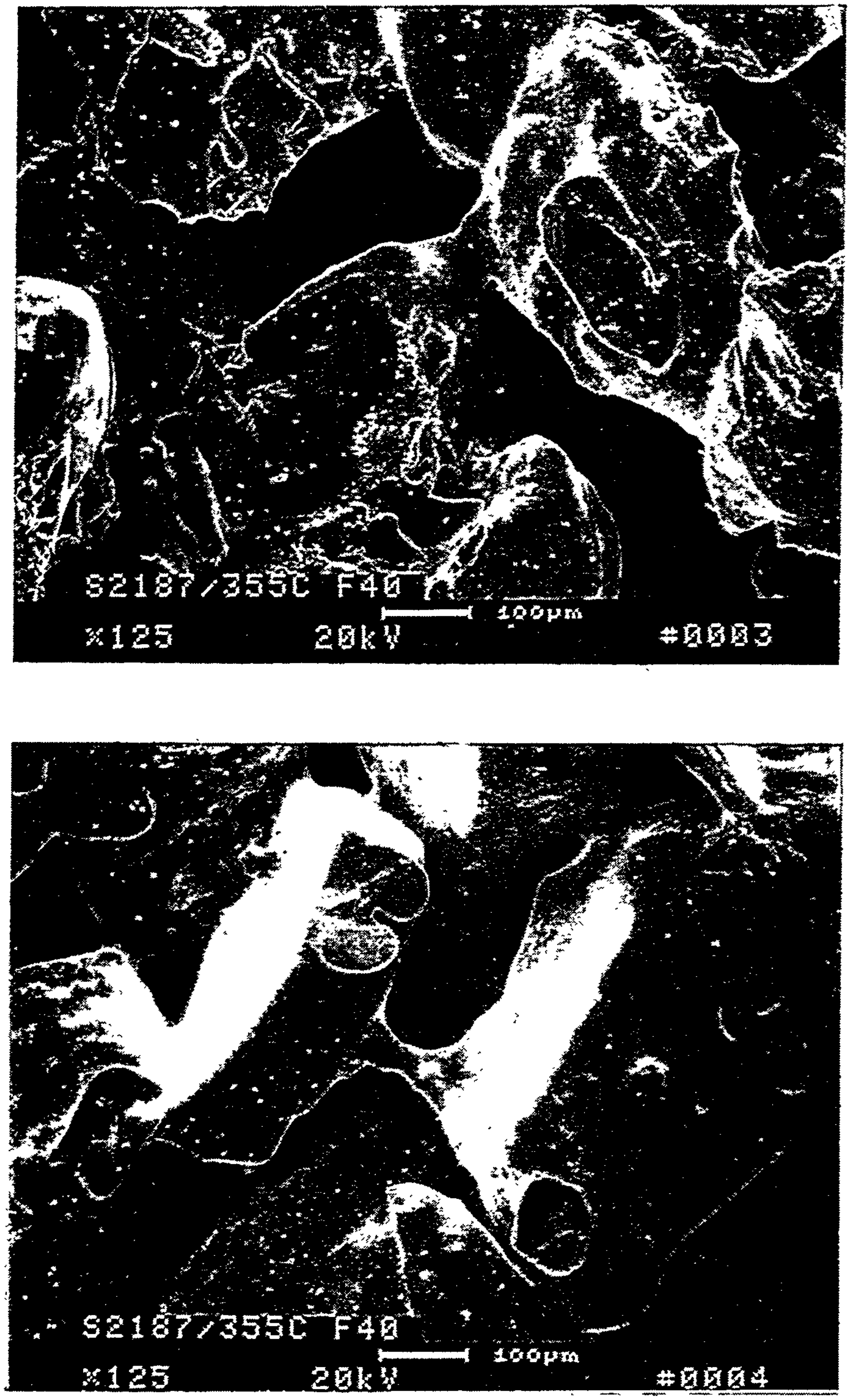


Figure 1b – Morphology Of The Coarse Support Silicon Carbide Support Structure In The Schumacher Dia Schumalith F40 Filter Matrix

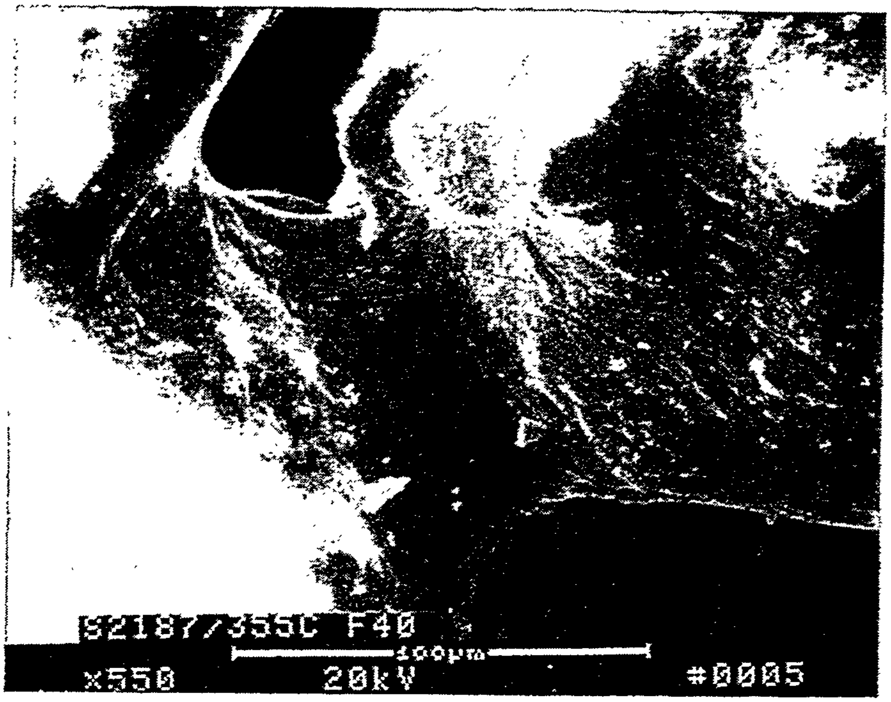


Figure 1c — Higher Magnification Micrograph Illustrating The Ligament Bond Post That Forms Between Adjacent Silicon Carbide Grains In The Schumacher Dia Schumalith F40 Filter Matrix

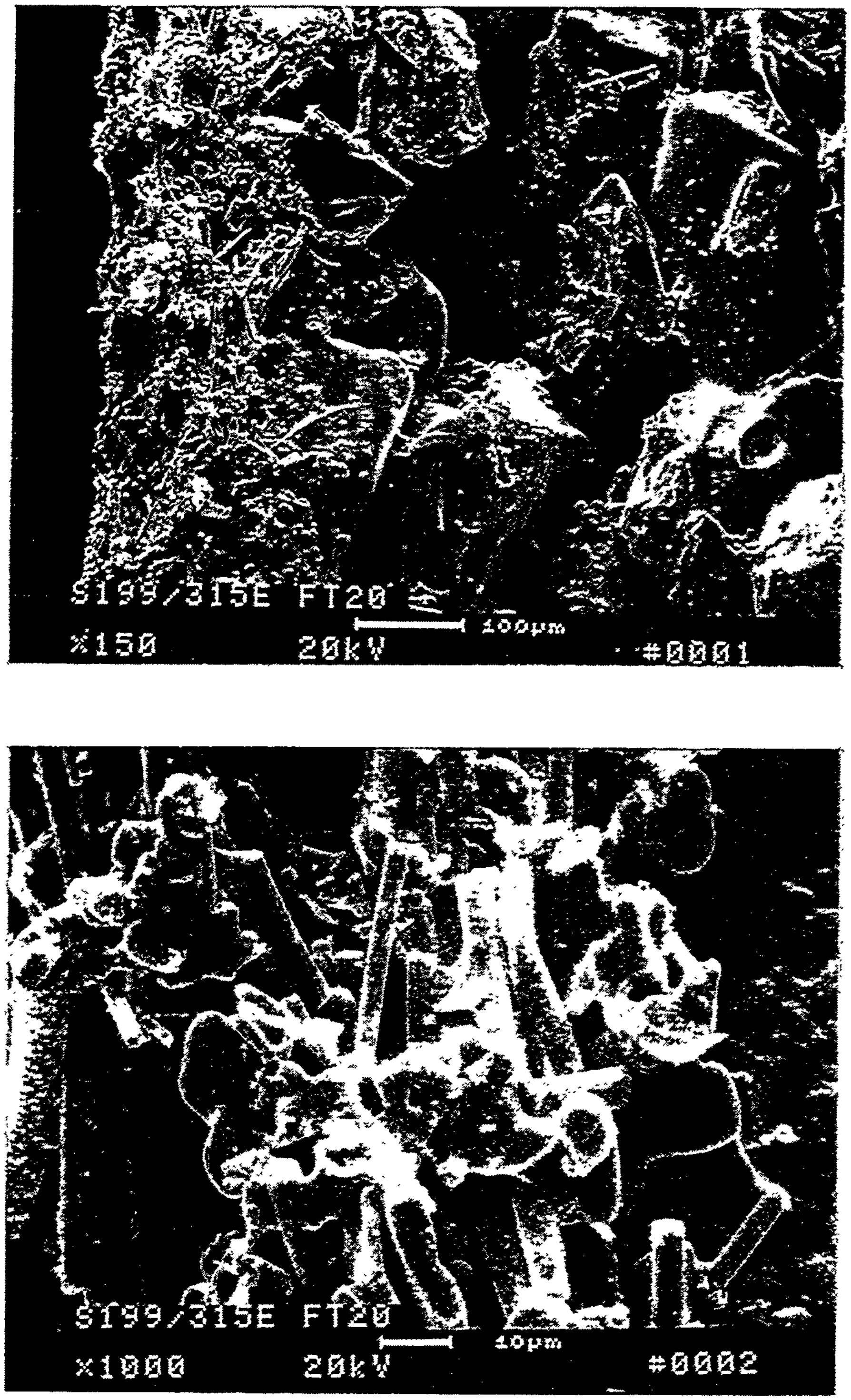


Figure 2a — Morphology Of The Cross-Sectioned Schumacher Dia Schumalith FT20 Filter Matrix Illustrating The Affinity Of The Aluminosilicate Fibrous Outer Membrane To The Underlying Support Silicon Carbide Grains

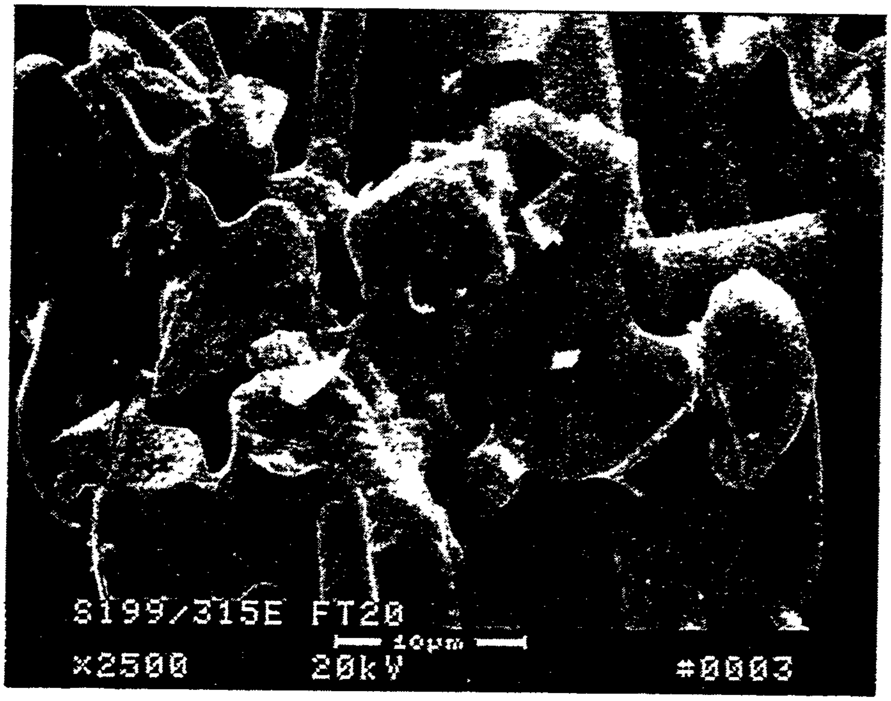


Figure 2b – Higher Magnification Micrograph Illustrating the Morphology Of The Aluminosilicate Fibers In The Schumacher Dia Schumalith FT20 Membrane

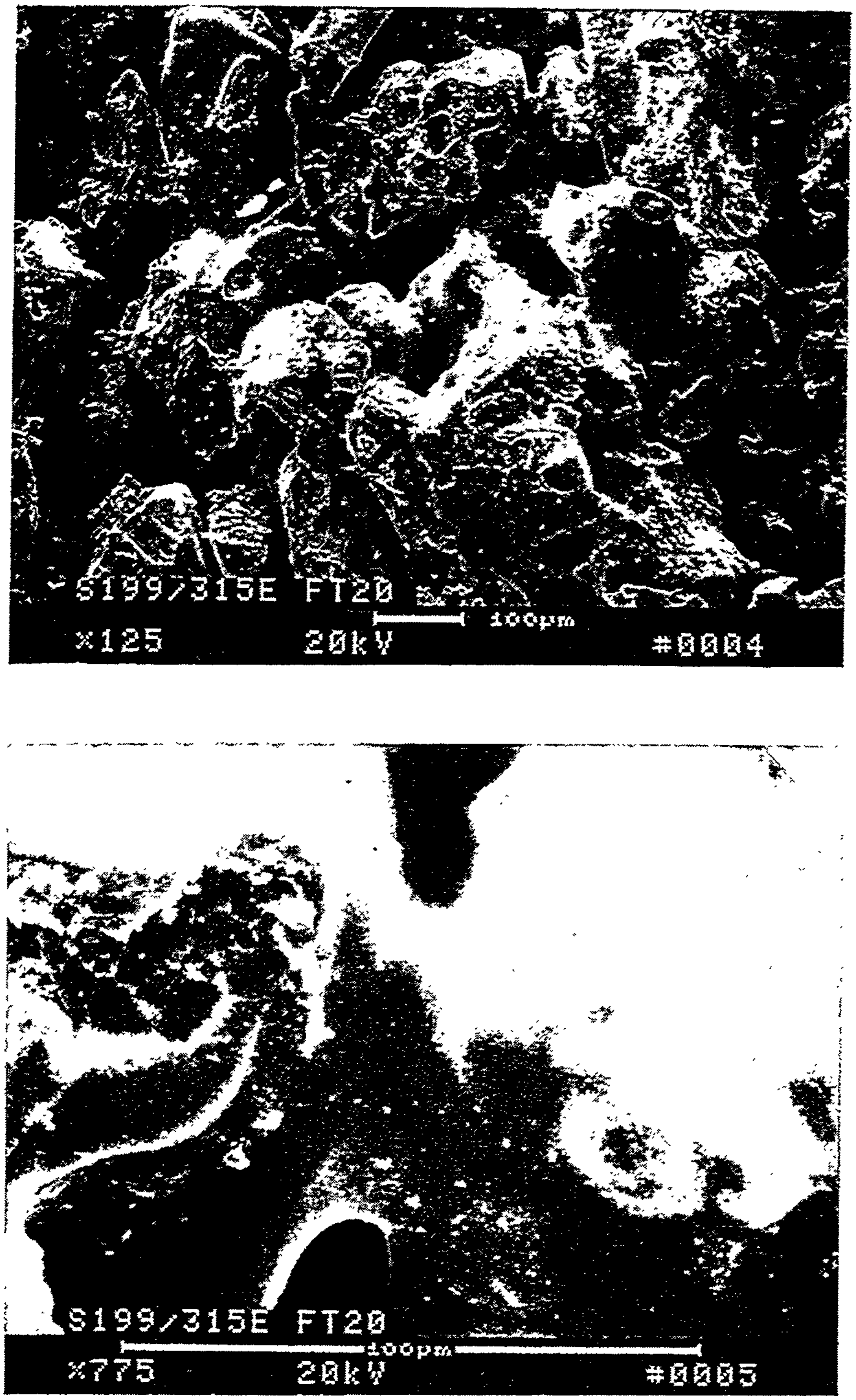


Figure 2c – Morphology Of The Silicon Carbide Grains In The Schumacher Dia Schumalith FT20 Filter Matrix

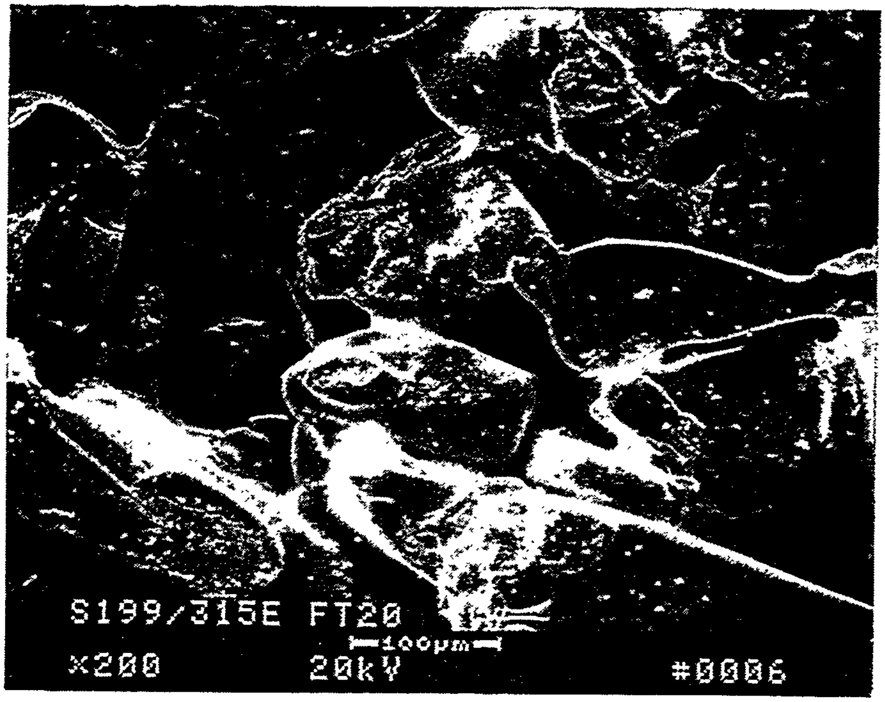


Figure 2d – Morphology Of The Silicon Carbide Grains In The Schumacher Dia Schumalith FT20 Filter Matrix

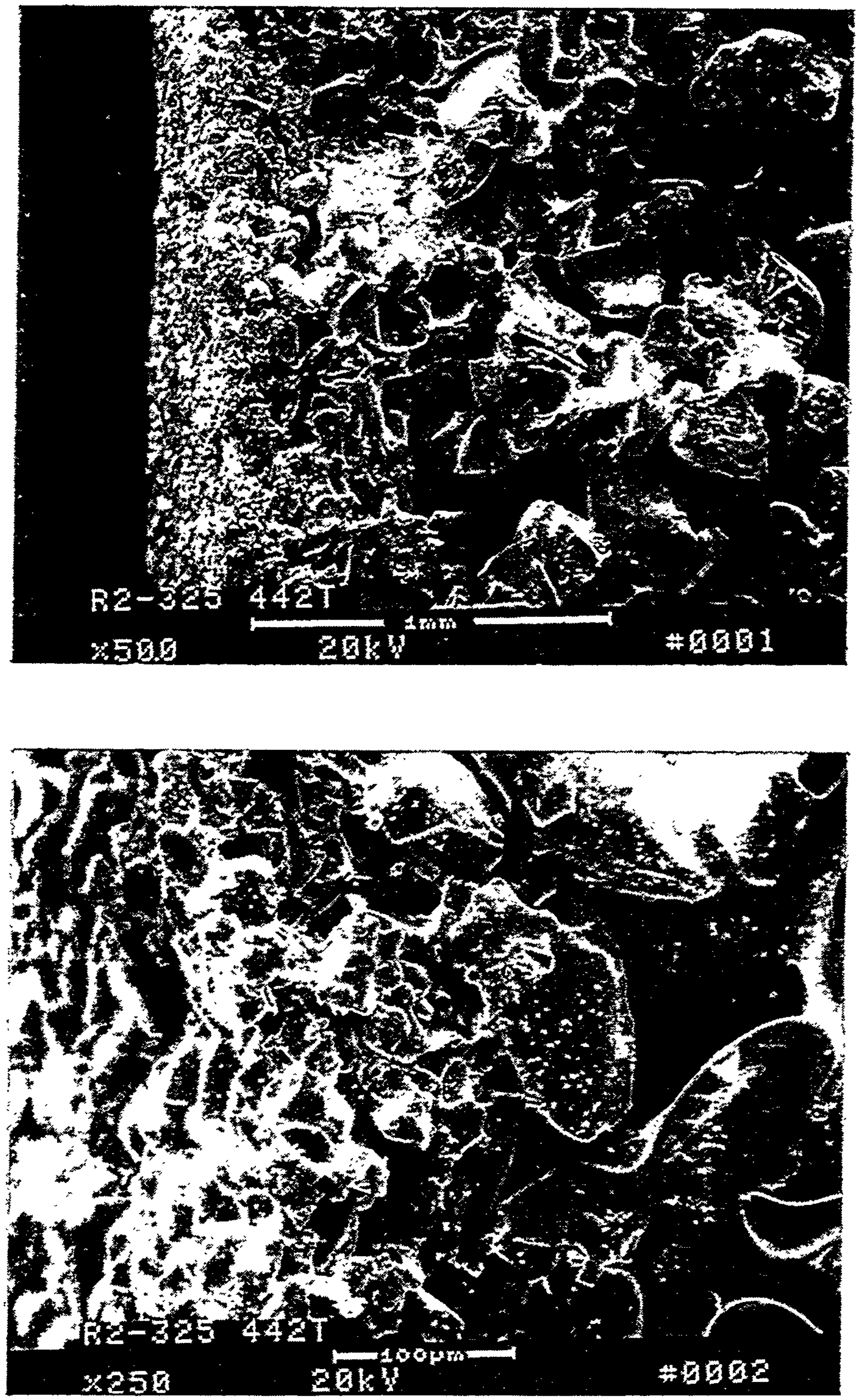


Figure 3a — Morphology Of The Cross-Sectioned Pall Vitropore 442T Filter Matrix Illustrating The Affinity Of The Finer Grained Silicon Carbide Outer Membrane To The Underlying Coarse Support Matrix

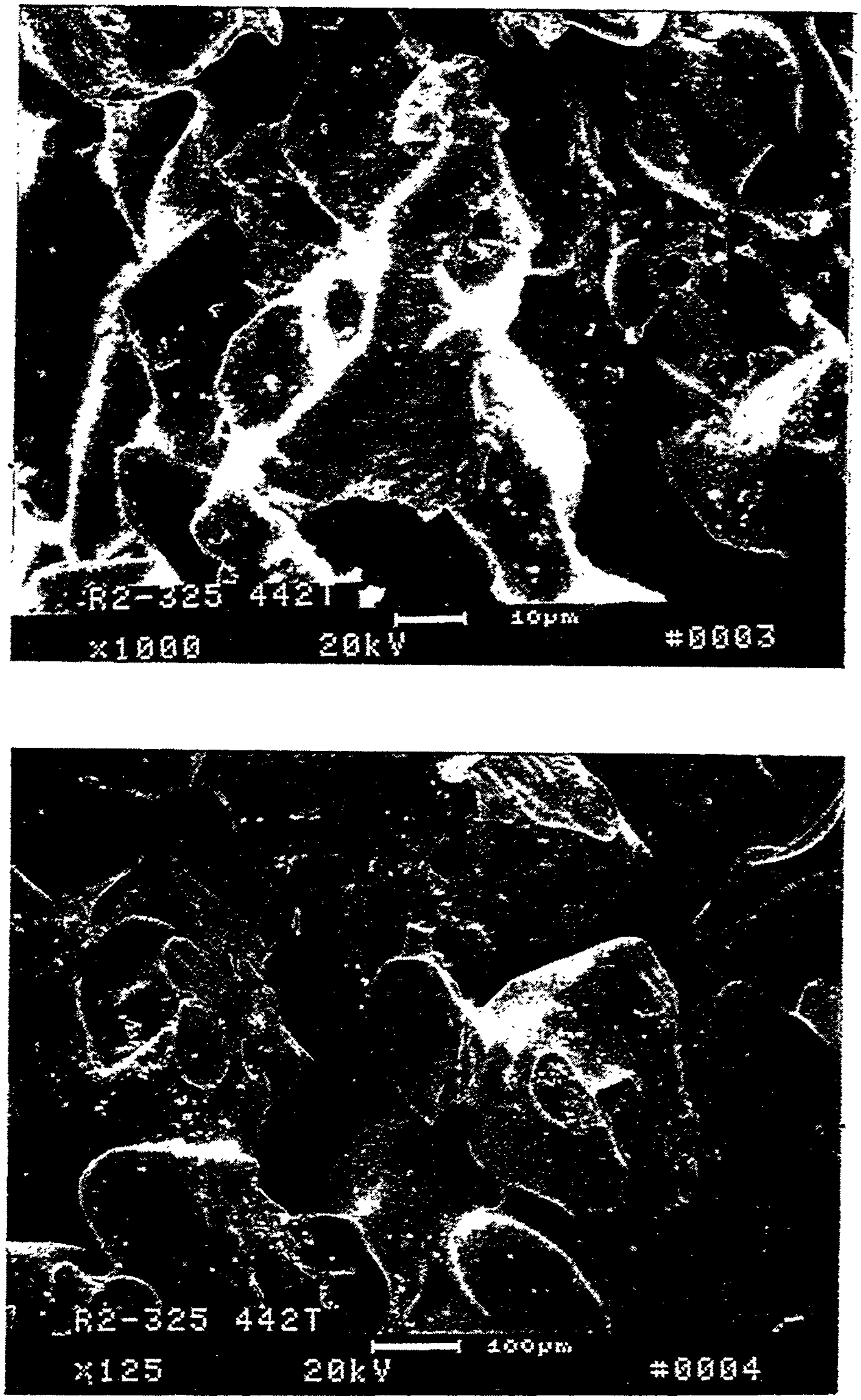


Figure 3b – Higher Magnification Micrograph Of The Silicon Carbide Grains In The Pall Vitropore 442T Filter Matrix (Top Photograph: Membrane; Bottom Photograph: Coarse Support Structure)

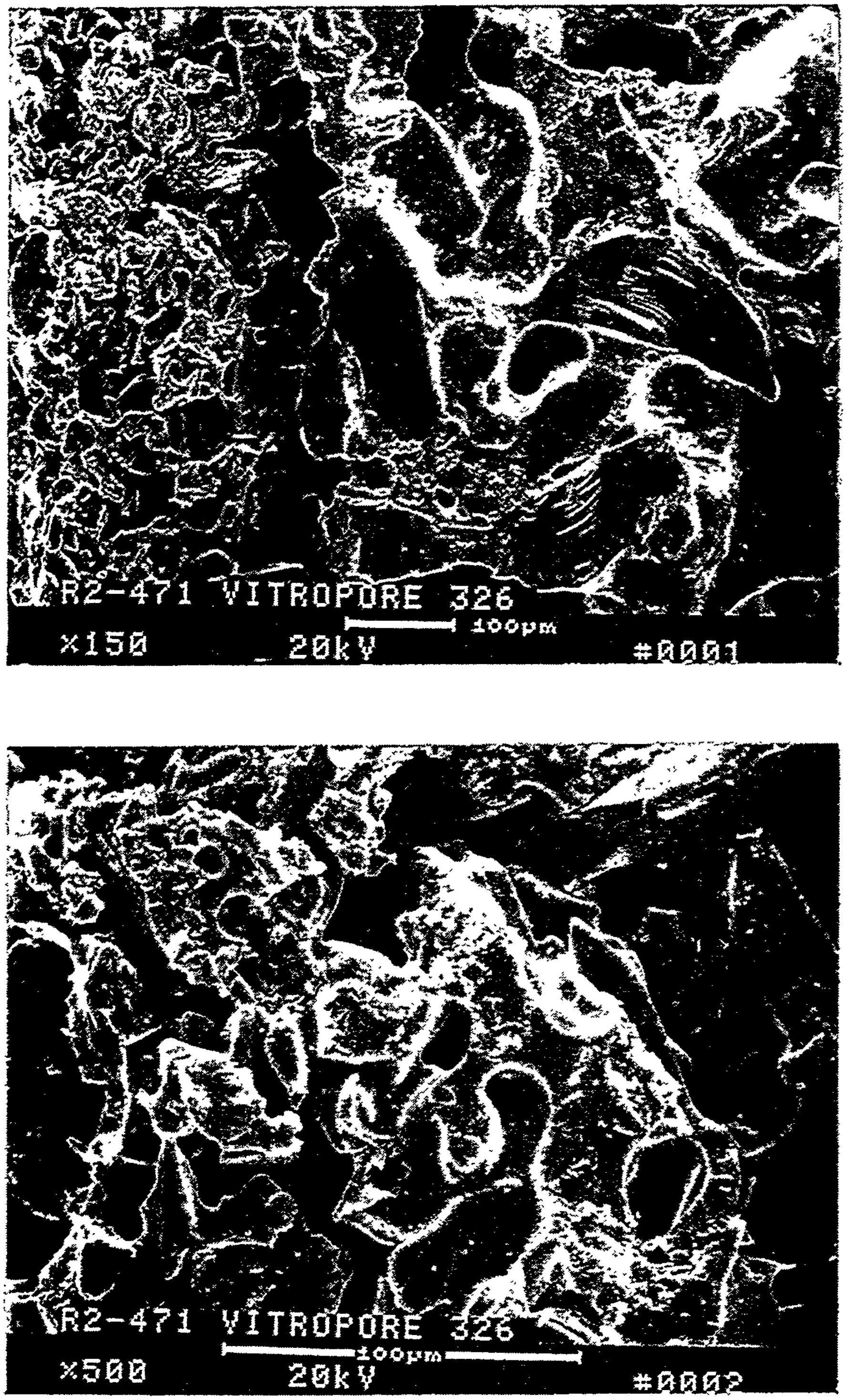


Figure 4a — Morphology Of The Cross-Sectioned Pall 326 Filter Matrix Illustrating The Affinity Of The Finer Grained Silicon Carbide Outer Membrane To The Underlying Coarse Support Matrix

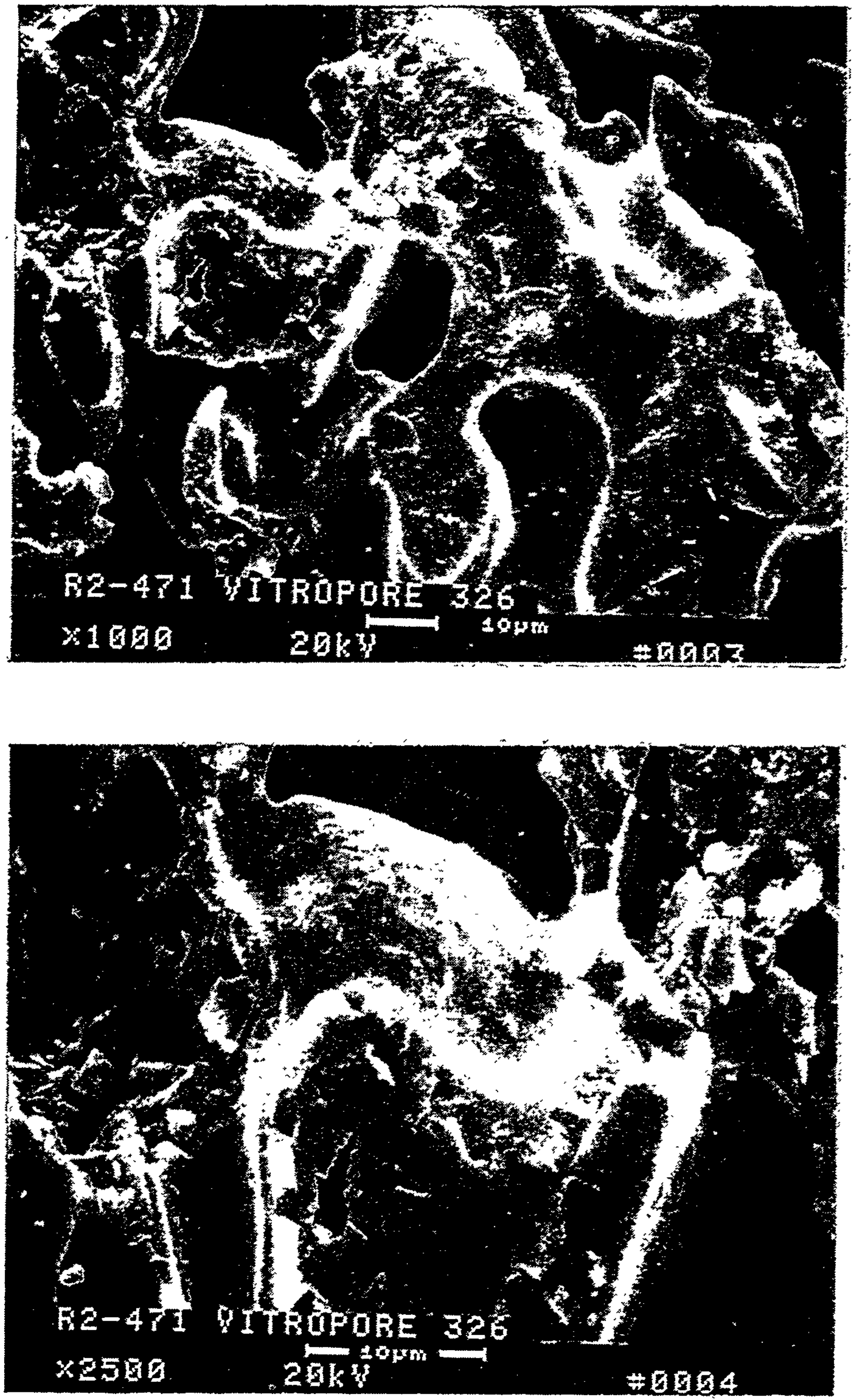


Figure 4b — Higher Magnification Micrographs Illustrating the Morphology Of The Silicon Carbide Grains In The Pall 326 Filter Membrane

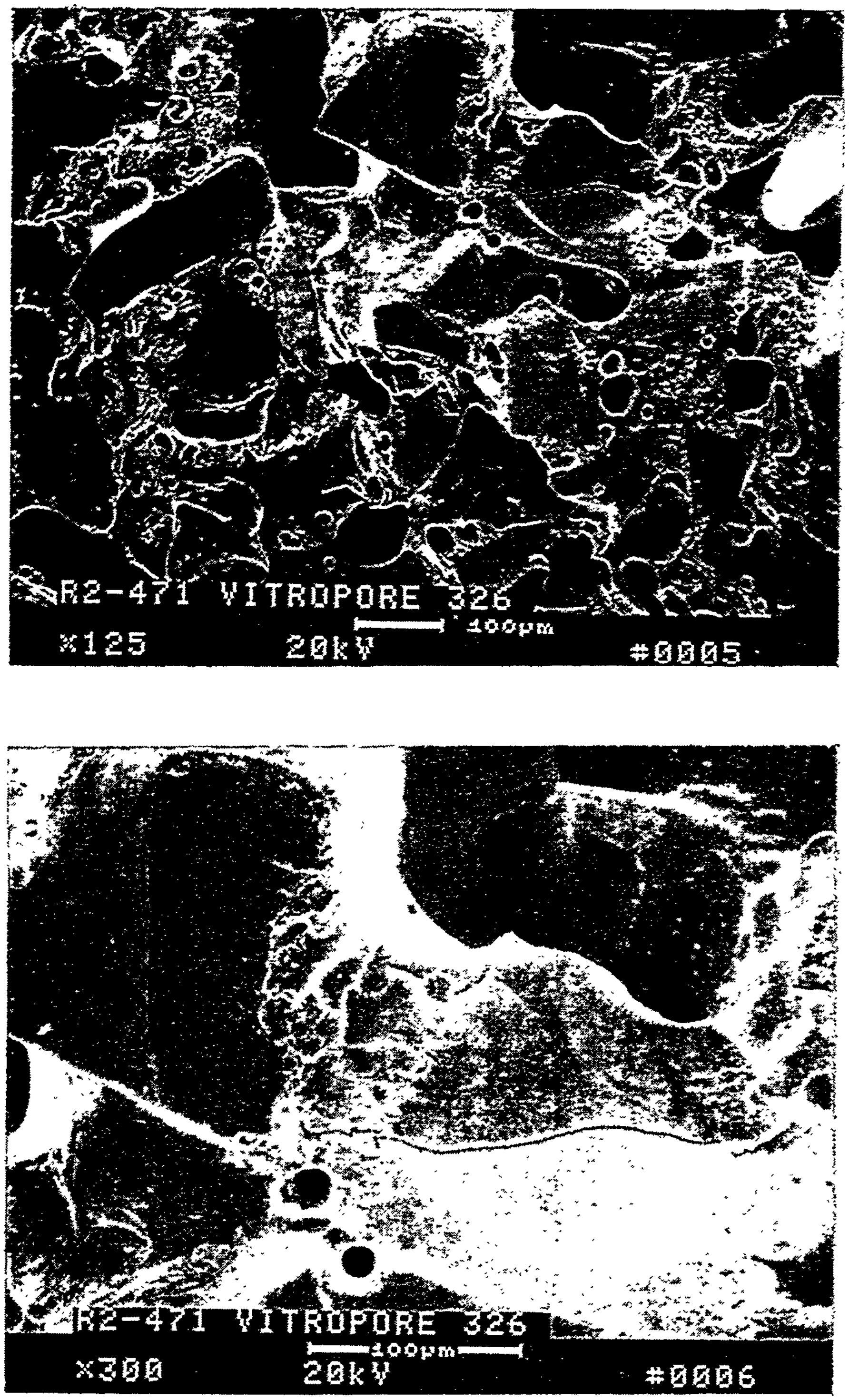


Figure 4c — Micrographs Illustrating The Binder Ligaments In The Fresh Fractured Pall 326 Filter Matrix Support Structure

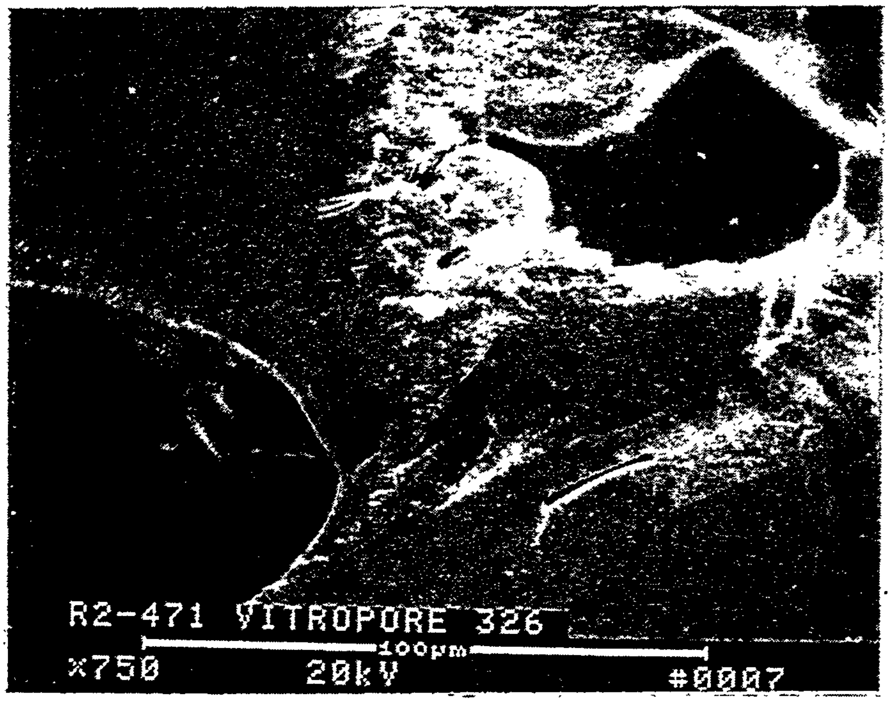


Figure 4d – High Magnification Micrograph Illustrating The Ligament Bond Post Between Two Adjacent Grains In The Coarse Support Structure Of The Pall 326 Filter Matrix

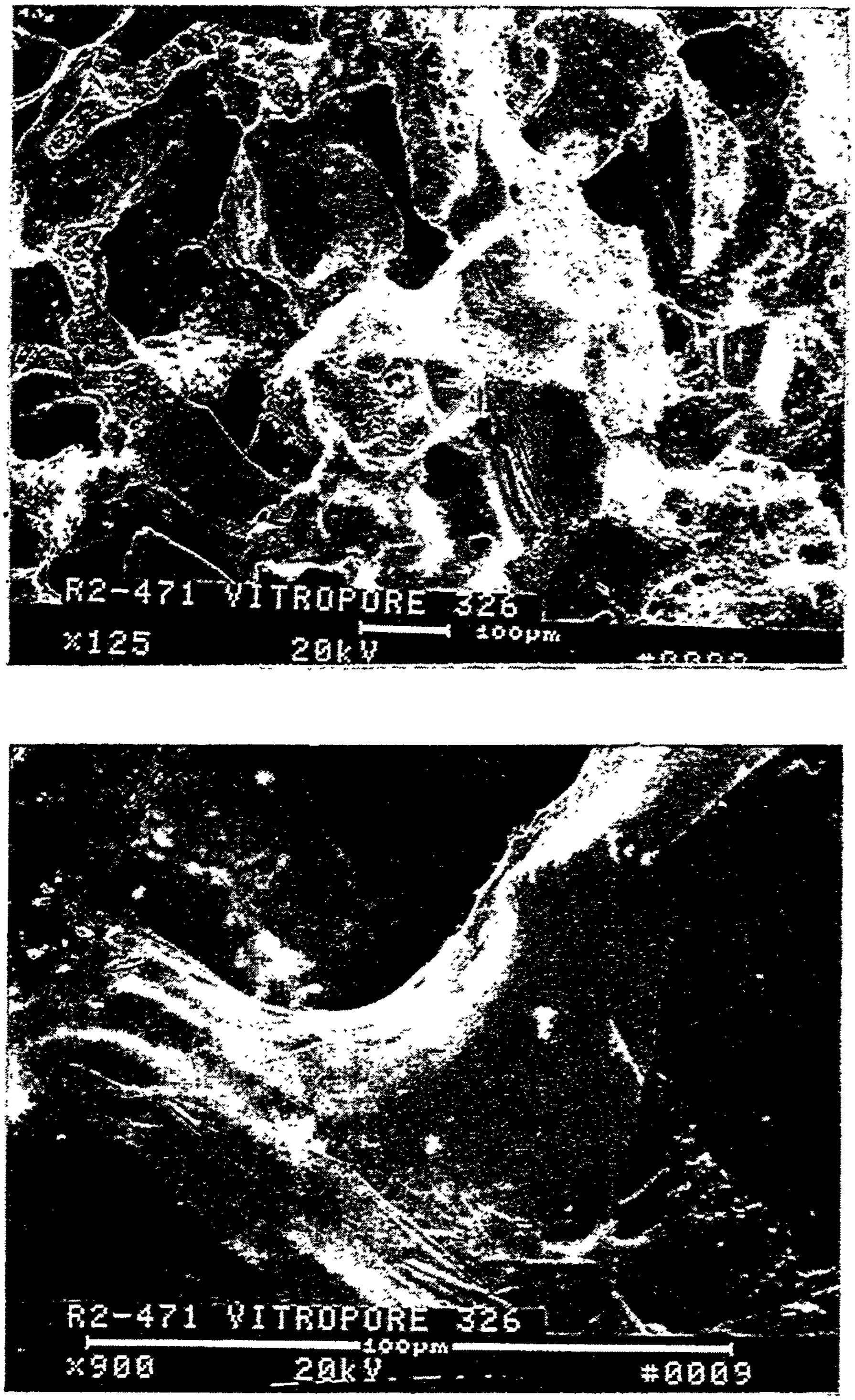


Figure 4e — Micrographs Illustrating The Morphology Of The Fresh Fractured Pall 326 Filter Matrix (Coarse Support Structure)

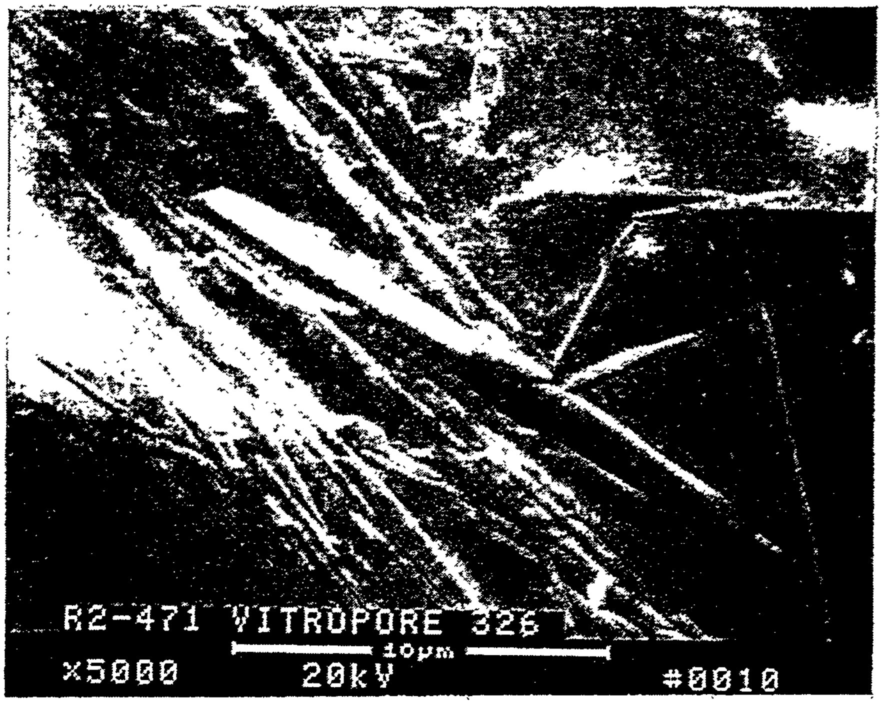


Figure 4f — Higher Magnification Micrograph Illustrating A Crystallized Area Along The Binder Surface Of The Ligament Bond Posts That Connects Two Adjacent Grains To Each Other

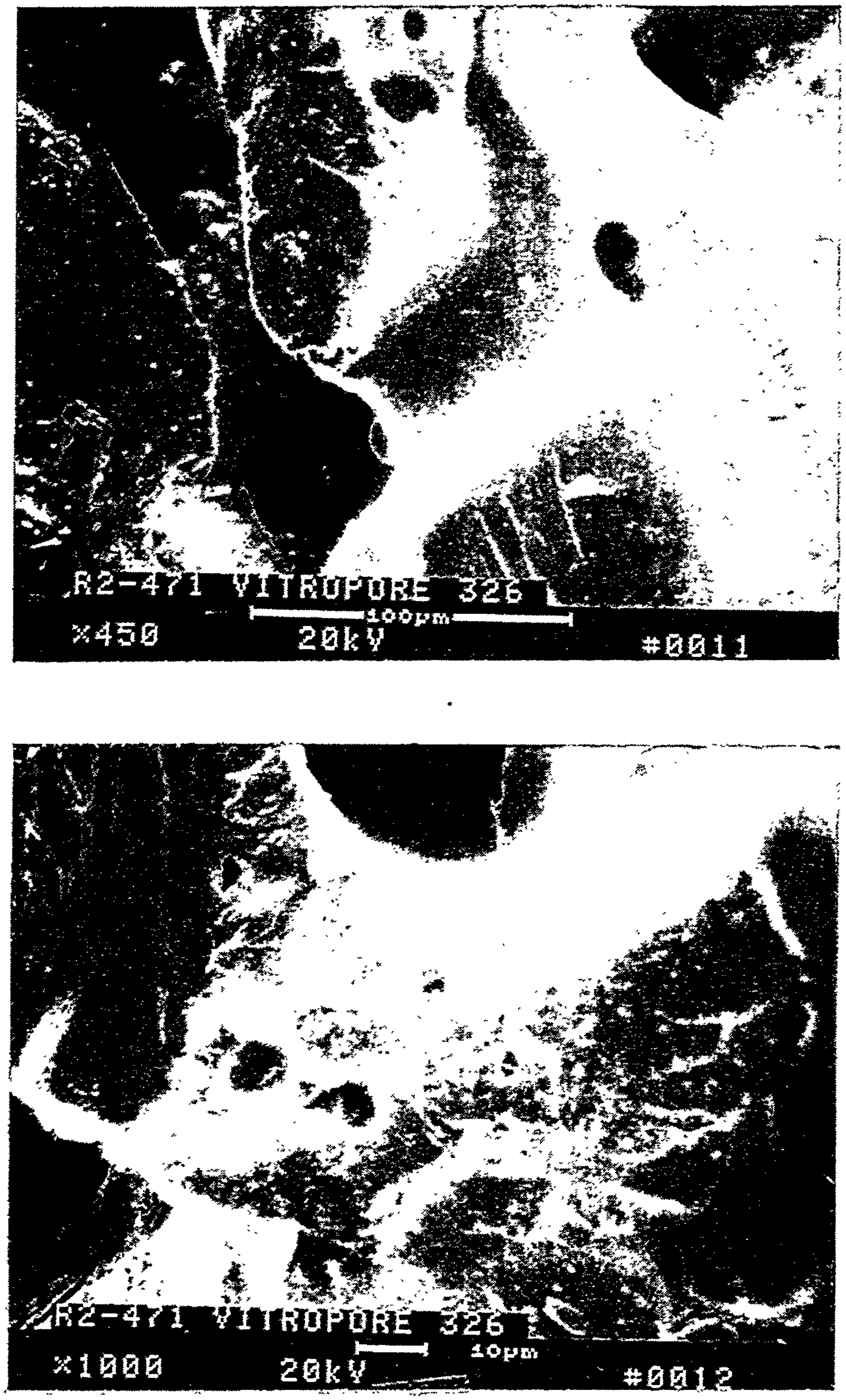


Figure 4g – Morphology Of The Fresh Fractured Binder Ligaments That Forms Between Adjacent Grains In The Support Structure Of The Pall 326 Filter Matrix. Void Formations Are Evident In The Fractured Ligaments.

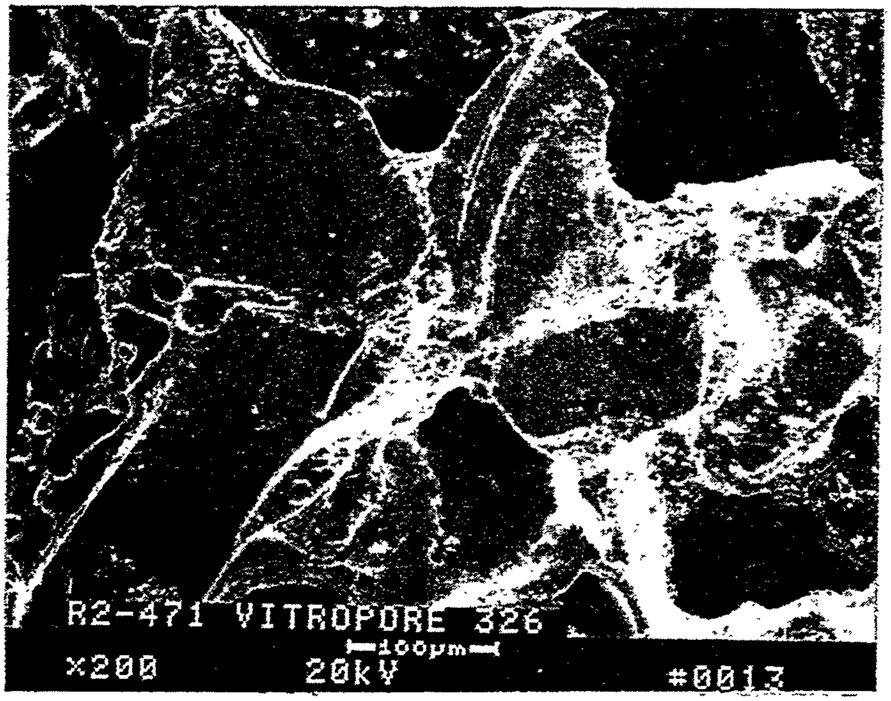


Figure 4h – Additional Micrograph Illustrating the Morphology Of The Fresh Fractured Pall 326 Coarse Support Wall

Energy dispersive x-ray analysis (EDAX) was utilized to semi-quantitatively determine the elemental composition of the binder phase in each filter material. Five areas were characterized in each matrix. The average and 1 σ atomic percent concentration for each element detected in the various filter materials is presented in Table 1.

The principle differences between the Schumacher Dia Schumalith F40 and FT20 filter matrices include

- A lower concentration of aluminum
- Possibly somewhat higher silicon concentration
- Lower sodium concentration
- Higher potassium concentration
- Lower iron concentration

in the Schumacher Dia Schumalith FT20 filter matrix. Similarly, titanium was removed from the Schumacher Dia Schumalith FT20 filter matrix.

In contrast, Pall's approach to producing an improved, high temperature, creep resistant filter material was to

- Increase the aluminum concentration
- Reduce the silicon concentration
- Add magnesium
- Reduce the sodium concentration
- Eliminate potassium and iron

in the 326 filter matrix.

In addition to the modified elemental composition of the binder, the proprietary, final high firing temperature and firing profile used at both candle supplier sites determine the binder phase composition which is initially present in the as-manufactured filter elements. Quantitative x-ray diffraction (XRD) analyses would be needed to identify the resulting phases in each material. The resulting phases are considered to directly impact the process temperature strength and creep resistance of the as-manufactured filter matrix.

TABLE 1

ELEMENTAL COMPOSITION ALONG THE SURFACE OF THE BINDER PHASE IN THE VARIOUS CLAY BONDED SILICON CARBIDE FILTER MATERIALS

Element	EDAX Atomic Percent							
Filter Matrix	O	Al	Si	Mg	Na	K	Fe	Ti
<u>Schumacher Dia Schumalith</u>								
F40	67.73±	5.74±	22.42±	---	2.07±	1.33±	0.57±	0.28±
S2187/355C *	6.87	1.52	6.38	---	0.09	0.65	0 22	0.13
FT20	65.35±	4.00±	26.79±	---	1.09±	2.29±	0.47±	---
S2199/315E	7.17	0.63	5.78	---	0.44	0.86	0.37	---
<u>Pall Vitropore</u>								
442T	58.99±	7.01±	27.55±	---	4.74±	0.94±	0.78±	---
R2-325	6.33	1.06	5.63	---	0.29	0.21	0.28	---
326	62.71±	8.35±	25.53±	1.24±	2.17±	---	---	---
R2-471	5.57	3.43	3.72	0.46	0.92	---	---	---

* Filter Identification Number.

As-Manufactured Filter Material Strength

The strength of the as-manufactured clay bonded silicon carbide filter materials was determined via C-ring compressive and tensile testing at room temperature, as well as at elevated temperatures. As shown in Table 2, depending on the manner in which the Schumacher material was fired, variable strengths could be obtained for the 1991 through 1993 production lots. Similarly by changing the coarse support silicon carbide grain size to a finer mesh, the strength of the filter matrix increased (i.e., Schumacher Dia Schumalith F40 vs FT20).

Depending on the manner in which Pall manufactured the Vitropore 442T filter matrix, variations in the as-manufactured strength of the filter element could be obtained. Currently Westinghouse has not determined the as-manufactured strength of the improved, high temperature, creep resistant Pall 326 candle filter matrix.

For the clay bonded silicon carbide filter materials, brittle fracture of the C-ring samples resulted at room temperature. The matrix retained its brittle fracture characteristics up to temperatures of ~900 °C (~1650 °F; Figure 5). Plastic deformation was initiated at higher compressive or tensile strength test temperatures. As the transition from brittle fracture to plastic deformation occurred, the resulting strength of the filter matrix decreased (Figure 6).

High Temperature Creep Testing

In the Ahlstrom PCFBC test facility in Karhula, Finland, elongation and catastrophic failure of the clay bonded silicon carbide Pall Vitropore 442T candle filter elements resulted after 500-1341 hours of operation at temperatures of 830 °C (1525 °F).(1,2) Examination of the flange area and fractured filter sections indicated that the clay bonded silicon carbide, Pall Vitropore 442T filter matrix experienced creep crack growth as the principal failure mechanism.

As a result, Westinghouse initiated a test effort which subjected the clay bonded silicon carbide filter materials to high temperature creep testing utilizing a four-point bend fixture. Test samples were 76 mm x 6.4 mm (3 in x 0.25 in) bend bars which were removed from candle filters. The bend bars were subjected to temperatures of 843 °C (1550 °F) for 300 hours with an applied load of 500 or 700 psi. Figure 7 indicates that

TABLE 2
AS-MANUFACTURED BULK STRENGTH OF THE CLAY BONDED SILICON CARBIDE CANDLE FILTER MATERIALS

Filter Matrix	Room Temp., Strength		Hot Strength (732 °C)	
	C-Ring Compression, psi	C-Ring Tension, psi	C-Ring Compression, psi	C-Ring Tension, psi
Schumacher Dia Schumalith F40				
1991	1285±197	---	1120±104 (a)	---
	1300±213	1907±111	1416±127	2328±228
1992	1790±112	2308±275	2064±204	3519±285
1993	1470±96	1971±367	1861±138	2381±271
	1451±83	1843±239	1450±100	2374±213
	1540±173	1821±266	1961±117	2026±351
	1451±83	1843±239	1450±100 (b)	2374±213 (b)
Schumacher 10/20				
1993	2296±261	2268±167	3043±148	2708±360
Pall Vitropore 442T				
Process No. 1	3152±130	2914±289	2749±351 (c)	2936±303 (c)
Process No. 2	3138±160	3387±271	2860±135 (d)	3176±280 (d)
Process No. 3	2585±202	2235±174	2406±247 (d)	2536±360 (d)
	2510±158	2654±256	2497±227 (b)	2730±238 (b)
Pall 326				
1995	TBD	TBD	TBD	TBD

(a) Strength Testing Conducted At 870 °C.
(b) Strength Testing Conducted At 843 °C.
(c) Strength Testing Conducted At 900 °C.
(d) Strength Testing Conducted At 830 °C.

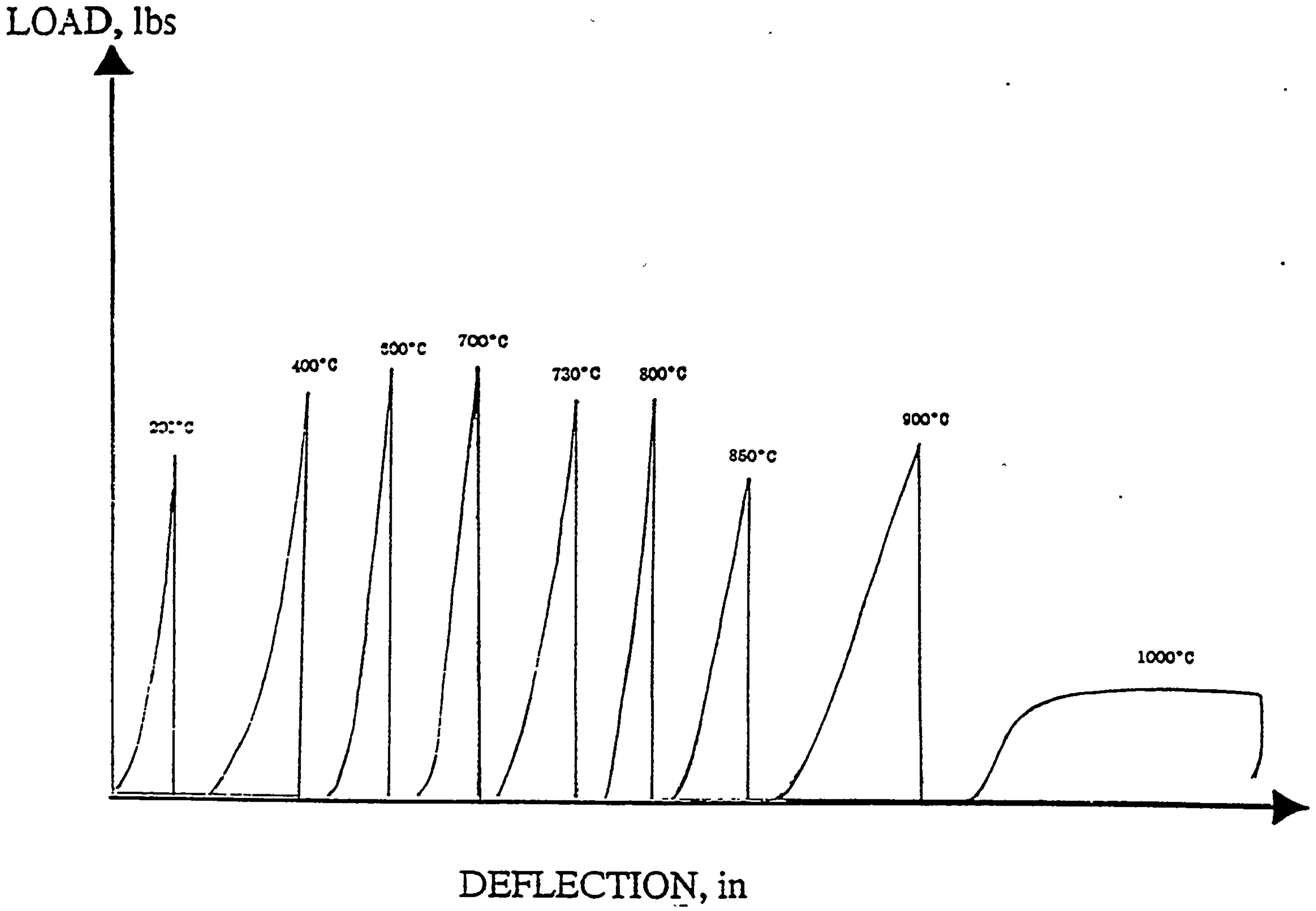


Figure 5 — C-Ring Compressive Load Versus Deflection Curves For The Schumacher Dia Schumalith F40 As-Manufactured Filter Matrix As A Function Of Temperature

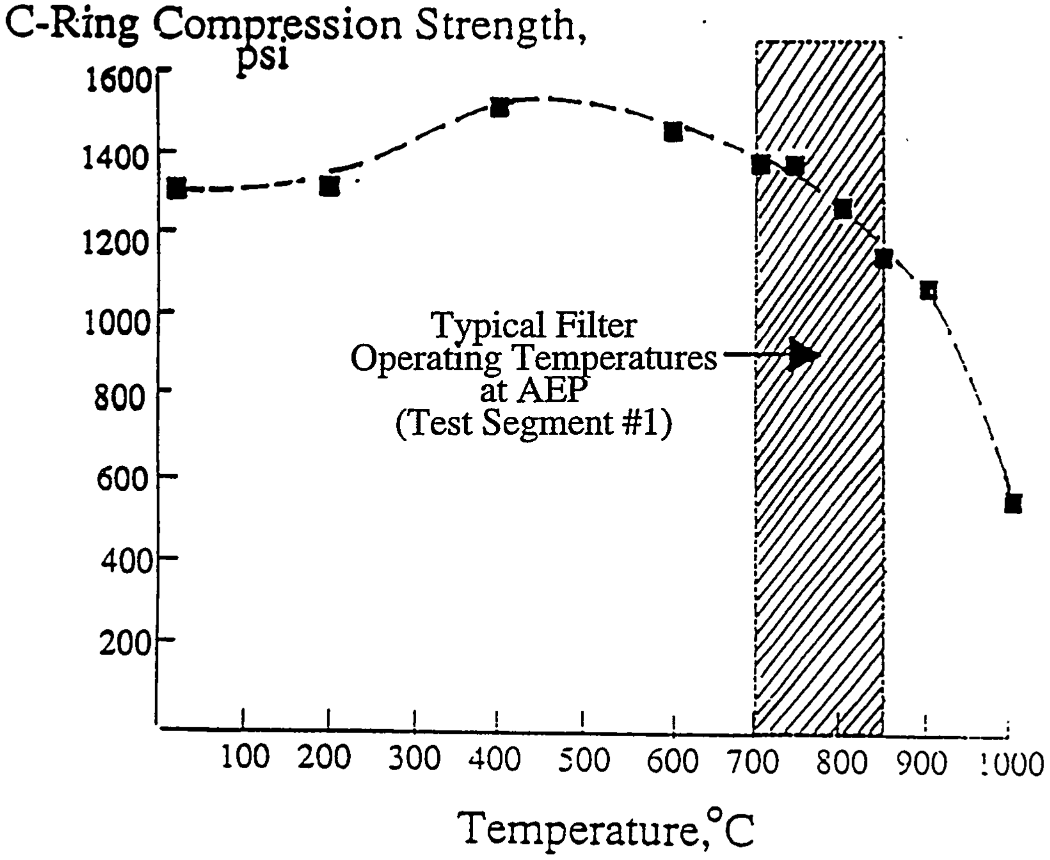


Figure 6 — C-Ring Compressive Strength Of The As-Manufactured Schumacher Dia Schumalith F40 Filter Matrix As A Function Of Temperature

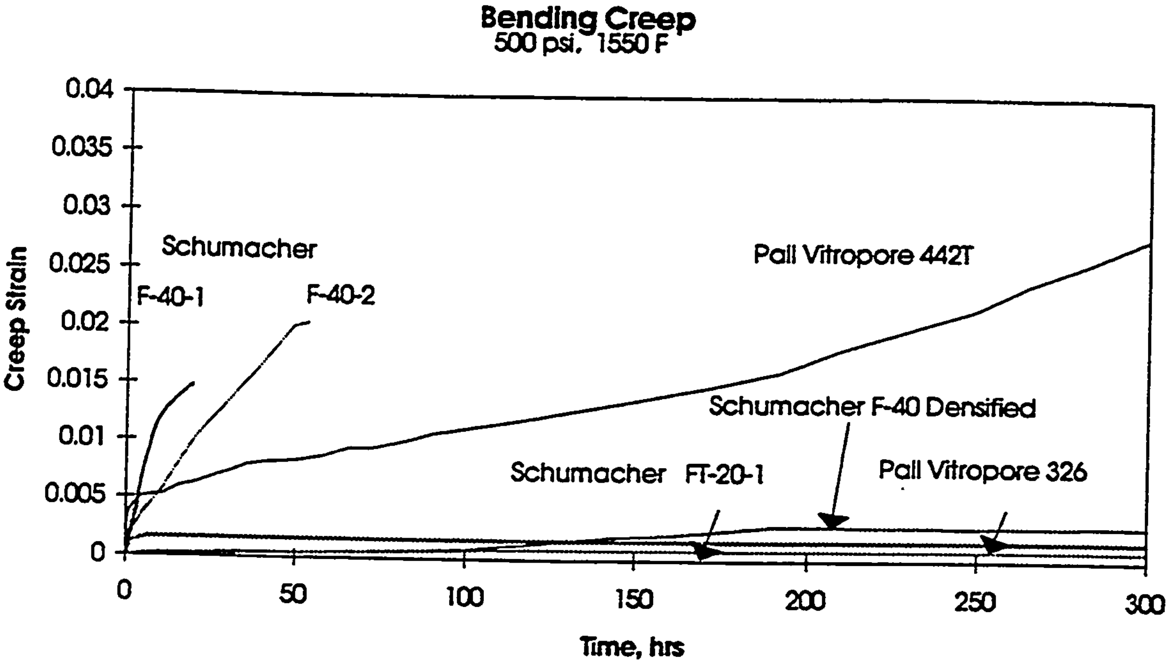


Figure 7 — High Temperature Creep Of The As-Manufactured Clay Bonded Silicon Carbide Candle Filter Materials

creep under these conditions results in the coarse matrix of the Schumacher Dia Schumalith F40 filter body, as well as in the Pall Vitropore 442T filters. Both of these materials consist of a clay binder which bonds adjacent silicon carbide grains to each other. At high temperature, the binder tends to soften, and under an applied load, both matrices experience creep.

Further efforts were also conducted using sections of material that were removed from the densified flange section of the clay bonded silicon carbide Schumacher Dia Schumalith F40 filter element. High temperature creep of the dense F40 matrix was shown to be limited in comparison to either the coarse Schumacher Dia Schumalith F40 matrix or the Pall Vitropore 442T matrix. An interesting observation which was made was that the smaller the grain size of the silicon carbide used to manufacture the filter matrix (i.e., greater number of ligament bond posts per unit volume), the higher the strength of the material, and the lower the high temperature creep.

Schumacher reformulated the binder phase of its Dia Schumalith F40 filter matrix, retained its traditional fibrous aluminosilicate membrane layer, but constructed the matrix with finer grit silicon carbide, producing what was considered by Schumacher to be a stronger, higher temperature, creep resistance filter matrix. When the Schumacher Dia Schumalith FT20 matrix was subjected to high temperature four-point bend creep testing at Westinghouse, the matrix demonstrated excellent creep resistance under the 500-700 psi applied load at temperatures of 843 ° C (1550 ° F).

As a result of the initial high temperature, bench-scale, creep test results, Westinghouse installed eight Schumacher Dia Schumalith FT20 filter elements at AEP in Test Segment #4. These filter were successfully operated for 1705 hours at nominal temperatures of 732 ° C (1350 ° F).

Pall has also produced an improved high temperature, creep resistant binder silicon carbide filter matrix. As shown in Figure 7, high temperature creep strain of the Pall 326 matrix was comparable to that of the Schumacher Dia Schumalith FT20 matrix, as well as the densified Schumacher Dia Schumalith F40 matrix.

HIGH TEMPERATURE, HIGH PRESSURE FILTER QUALIFICATION TESTING

Filter qualification testing has been conducted at the Westinghouse Science and Technology Center, high temperature, pressurized fluidized-bed combustion (PFBC) test facility in Pittsburgh, PA. Qualification testing is generally performed on advanced filter elements and/or concepts prior to installation and operation of the filters in the field. This section reviews the results of the qualification tests which have been conducted utilizing the advanced Pall 326 and Schumacher 10/20 candle filters.

Pall 326 Candle Filter Testing

An improved, high temperature, creep resistant Pall 326 candle was included in a filter array that was subjected to thermal fatigue testing (i.e., accelerated pulse cycling) in Westinghouse's HTHP PFBC test facility. Testing was conducted at temperatures of 843 °C (1550 °F) and pressures of 100 psig. A cold pulse of air was delivered to the candle filter array every 2.5 minutes. The thermal duty of the pulse cleaning cycle in the HTHP PFBC test facility was 50 Btu/pulse-ft^2 which was comparable to the thermal duty of the pulse cleaning gas that was utilized in the Westinghouse Advanced Particulate Filtration (W-APF) system which was installed and operated at the American Electric Power (AEP) Demonstration Plant in Brilliant, OH, between October 1992 and March 1995.

During HTHP PFBC testing at Westinghouse, the newly manufactured Pall 326 candle filter acquired 1860 accelerated pulse cleaning cycles, prior to test termination. Post-test inspection of the Pall 326 filter element indicated that the candle remained intact, and did not experience any visible bowing and/or elongation during the ~78 hour, HTHP PFBC qualification test.

Additional material characterization (i.e., destructive strength testing) has not been conducted to date on the accelerated pulse cycled, Pall 326 filter element.

Schumacher 10/20 Candle Filter Testing

Advancements have been made to provide increased high temperature strength in the Schumacher clay bonded silicon carbide candle filters. Preliminary testing conducted at Westinghouse characterized the advanced Schumacher 10/20 clay bonded silicon carbide filter matrix both in terms of initial as-manufactured material strength, as well as residual strength after being subjected to accelerated high temperature, thermal fatigue testing. Our results indicated that although the matrix initially had a higher room temperature and process temperature strength in comparison to the Schumacher Dia Schumalith F40 filter matrix, the Schumacher 10/20 matrix lost strength, similar to what had been demonstrated for the Schumacher Dia Schumalith F40 filter elements. Unlike the 15 mm Schumacher Dia Schumalith F40 wall, the 10 mm Schumalith 10/20 matrix experienced significant crack formations along its inside surface after being subjected to 10,000 accelerated thermal pulse cycles. Although the Schumacher 10/20 matrix lost strength and contained ID cracks, the candle filter remained intact after being subjected to thermal fatigue testing in Westinghouse's HTHP PFBC test facility.

HTHP Filter Testing

Three 1.5 m porous ceramic clay bonded silicon carbide filters were received from Schumacher GmBh in March 1994. The identification numbers of the three candles were S52/342D, S65/342D, and S67/342D. The matrix was designated as 10/20 and consisted of silicon carbide grains that were bonded together via the standard clay binder used in the manufacture of the Schumacher Dia Schumalith F40 matrix. Unlike the Schumacher Dia Schumalith F40 filters, the Schumacher 10/20 filters were manufactured with a 10 mm wall (vs 15 mm), and an alumina-based granulated OD membrane (vs aluminosilicate fibrous membrane).

HTHP PFBC testing consisted of exposing two Schumacher 10/20 filter elements with six alternate porous ceramic candle filters to steady state operating conditions (i.e., 843 °C (1550 °F) and 100 psi) between March 14, 1994 and April 16, 1994 (i.e., two test segments which were operated continuously on a 24 hour/day basis).(3) Ten thousand, 0.4 sec cold pulse cycles were delivered to the candle array at approximately 1.5 minute intervals throughout the entire course of testing. During HTHP testing, the Westinghouse fail-safe/regenerator device was installed above candle filter S67/342D in order to preheat the incoming cold pulse gas. The thermal duty of the cold pulse was comparable to that used in the Westinghouse Advanced Particulate Filtration (W-APF) system in the American Electric Power (AEP) demonstration plant in Brilliant, Ohio. After every 500 pulse cycles, Grimethorpe PFBC ash was delivered to the candle filter array for 15 minutes in order to determine whether the filter array was intact.

After completion of the 10,000 accelerated pulse cycle exposures, the vessel was slow cooled and the array was lifted and inspected. The Schumacher 10/20 candles were observed to have remained intact during high temperature, accelerated thermal fatigue testing. Further post-test characterization included strength testing of the accelerated pulse cycled filters (i.e., room temperature and process temperature C-ring compressive and tensile testing), as well as identification of microcrack or crack formations through the accelerated pulse cycled 10/20 matrix. The results of these analyses follow.

Material Characterization

The bulk compressive and tensile strengths of the as-manufactured and accelerated pulse cycled Schumacher 10/20 filter matrix are shown in Table 3. The strengths of the Schumacher Dia Schumalith F40 candle filters which were also exposed during the same test effort are also provided in Table 3. The room temperature strength of the as-manufactured Schumacher 10/20 matrix is approximately twice that of the Schumacher Dia Schumalith F40 matrix. Similarly, the process temperature strength of the as-manufactured Schumacher 10/20 matrix is approximately 1.5 times that of the Schumacher Dia Schumalith F40 process temperature strength.

After exposure to 10,000 accelerated pulse cleaning cycles, the room temperature strength of the Schumacher 10/20 filter matrix decreased by 24-27% along its OD surface (C-ring compressive testing), and by 29-37% along its ID surface (C-ring tensile testing) surface. The bulk strength of the pulse cycled Schumacher 10/20 filter matrix at process operating temperatures was, however, observed to decrease by only 12-14% along its OD surface, and by 1-7% along its ID surface.

Table 3

Strength Characterization of Thermally Fatigued Porous Ceramic Filter Materials

Filter Identification Number	Test No.	Accelerated Pulse Cycles	Fail-Safe/ Regenerator	Room Temp., Strength C-Ring Compression, psi	Room Temp., Strength C-Ring Tension, psi	Hot Strength,(843°C) C-Ring Compression, psi	Hot Strength,(843°C) C-Ring Tension, psi	Burst Strength, psi
SCHUMACHER DIA SCHUMALITH 10/20 CLAY BONDED SILICON CARBIDE								
S65/342D	--	---	---	3236±303	3588±376	3024±188	3639±253	TBD
S52/342D	1	10,000	No	2466±105	2260±202	2661±295	3587±258	TBD
S67/342D	2	10,000	Yes	2363±155	2560±265	2601±210	3396±209	TBD
SCHUMACHER DIA SCHUMALITH F40 CLAY BONDED SILICON CARBIDE								
S2097/352C	--	---	---	1451± 83	1843±239	1450±100	2374±213	TBD
S2102/352C	3	771	No	1393±147	1574±381	1217±120	1344± 60	TBD
S2099/352C	1	10,000	No	1244± 57	1466±156	1204± 51	2202±345	TBD
S2104/352C	4	713	Yes	1497±127	1338± 93	1238± 80	1359± 76	TBD
S2103/352C	2	10,000	Yes	1242± 74	1142±125	1323±106	1610± 82	TBD

In contrast, a 14% and 21-38% reduction in the room temperature strength resulted along the OD and ID surfaces, respectively, of the Schumacher Dia Schumalith F40 filter matrix after exposure to 10,000 accelerated pulse cleaning cycles. At process operating temperatures, a strength reduction of 9-17% and 7-32% resulted along the OD and ID surfaces of the filter element, respectively.

Microcrack Formation

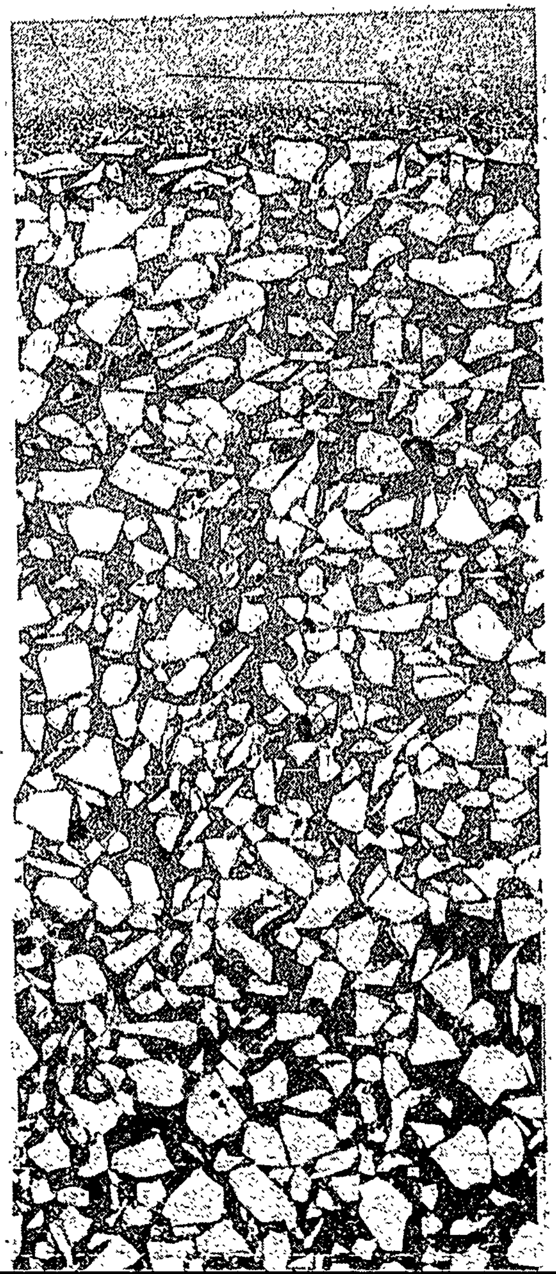
Several O-ring sections which were removed from the accelerated pulse cycled Schumacher 10/20 candles were evaluated in terms of possible microcrack formations. The O-rings were mounted in a Hysol epoxy mount and diamond polished. Optical micrograph montages were generated across wall thickness of O-rings which had been removed from the top, center, and bottom areas of candle filter S52/342D (Figure 8; absence of the fail-safe/regenerator unit), and from an O-ring that was removed from candle filter S67/342D (Figure 9; presence of fail-safe/regenerator unit).

As shown in Figure 8, circumferential crack formations were evident at ~0.4-0.6 mm from the ID surface of O-rings removed from the top and middle section of candle filter S52/342D (i.e., absence of the fail-safe/regenerator). The very evident circumferential crack pattern was not apparent along the O-ring section removed from the bottom of candle S52/342D. Similar crack formations were not readily evident in candle S67/342D matrix (i.e., fail-safe/regenerator installed above the filter element). This appears to be consistent with reduced thermal fatigue that is expected to occur with the use of the regenerator device. Further characterization is, however, needed to discern whether crack formations are a random phenomena which occur along the length of the 1.5 m clay bonded silicon carbide candle filters which had been subjected to extended accelerated pulse cycling with/without the use of the Westinghouse fail-safe/regenerator device.

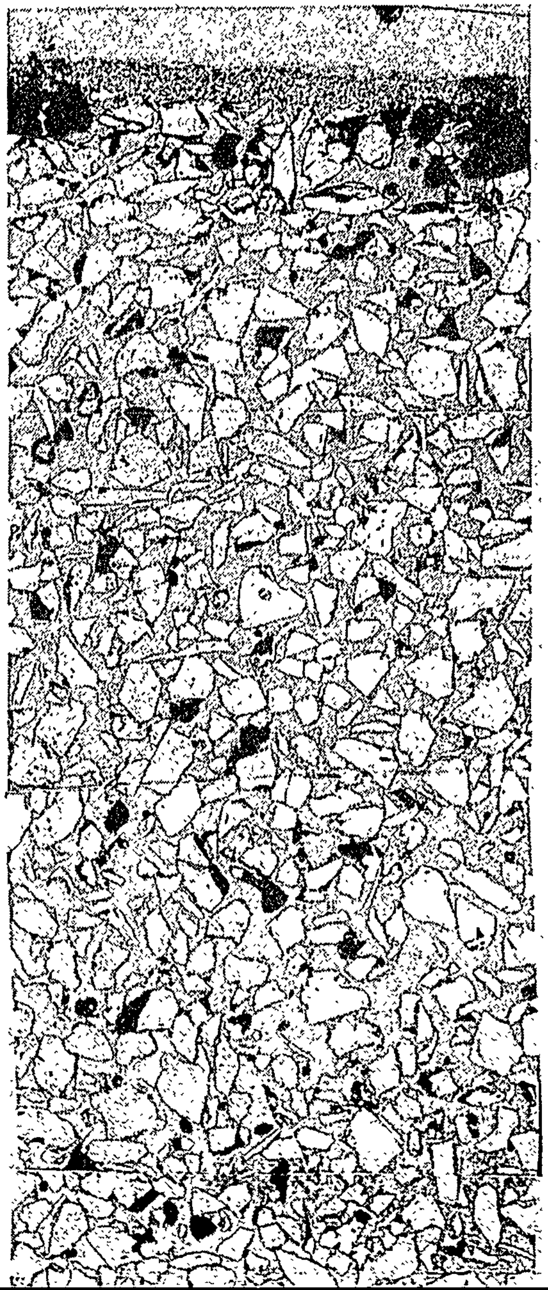
The presence of cracks along the candle filter ID wall implies the potential for additional microcracks to be present within the filter matrix wall. Detection of microcracks is extremely difficult within the highly porous clay bonded silicon carbide filter matrix. The potential existence of microcracks, as well as the obvious ID crack formations support the reported reduction in material strength after extended exposure to accelerated, high temperature, pulse cycling conditions.

S52/342D (Top)

S52/342D (Center)



S52/342D (Bottom)



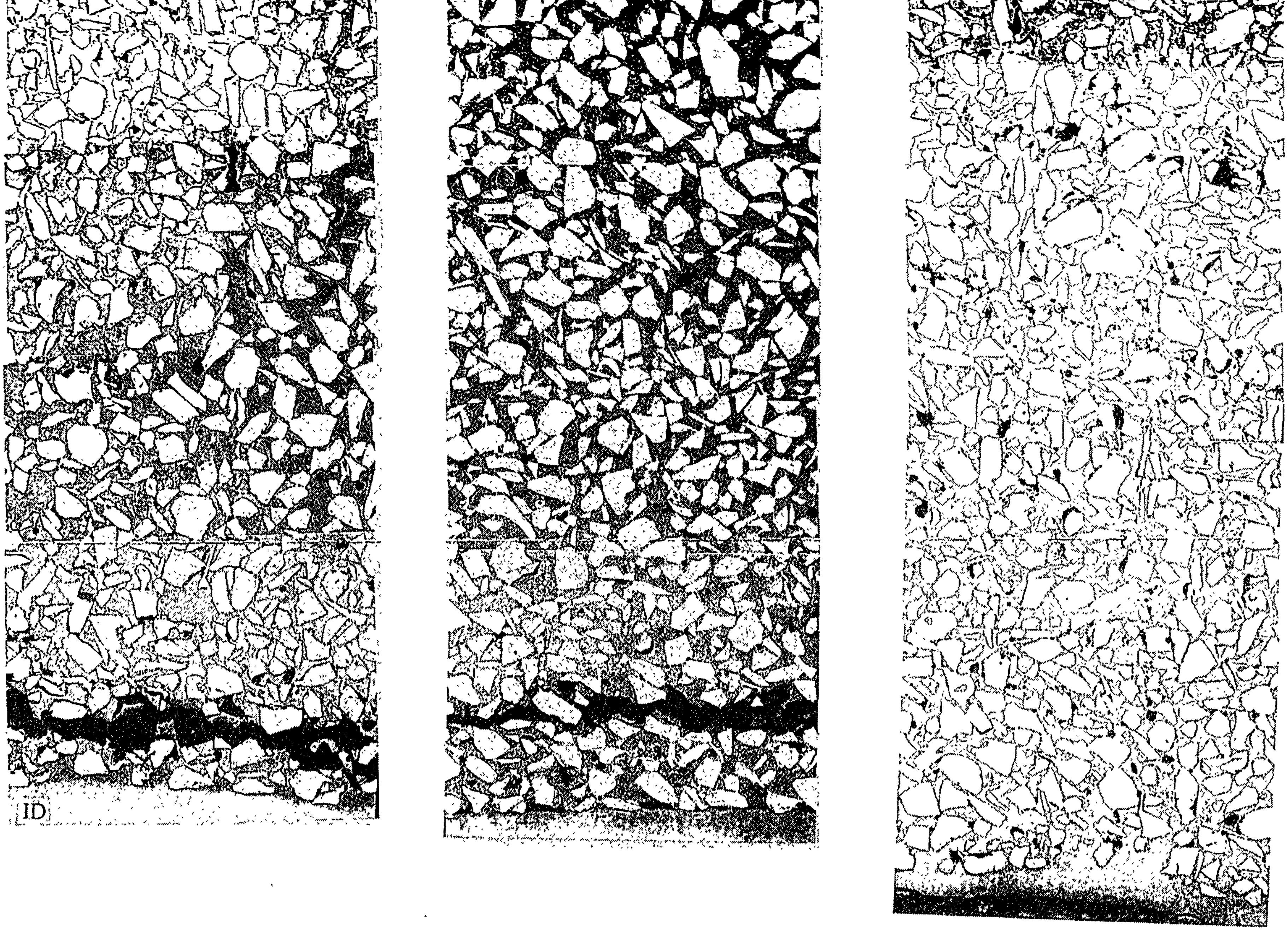
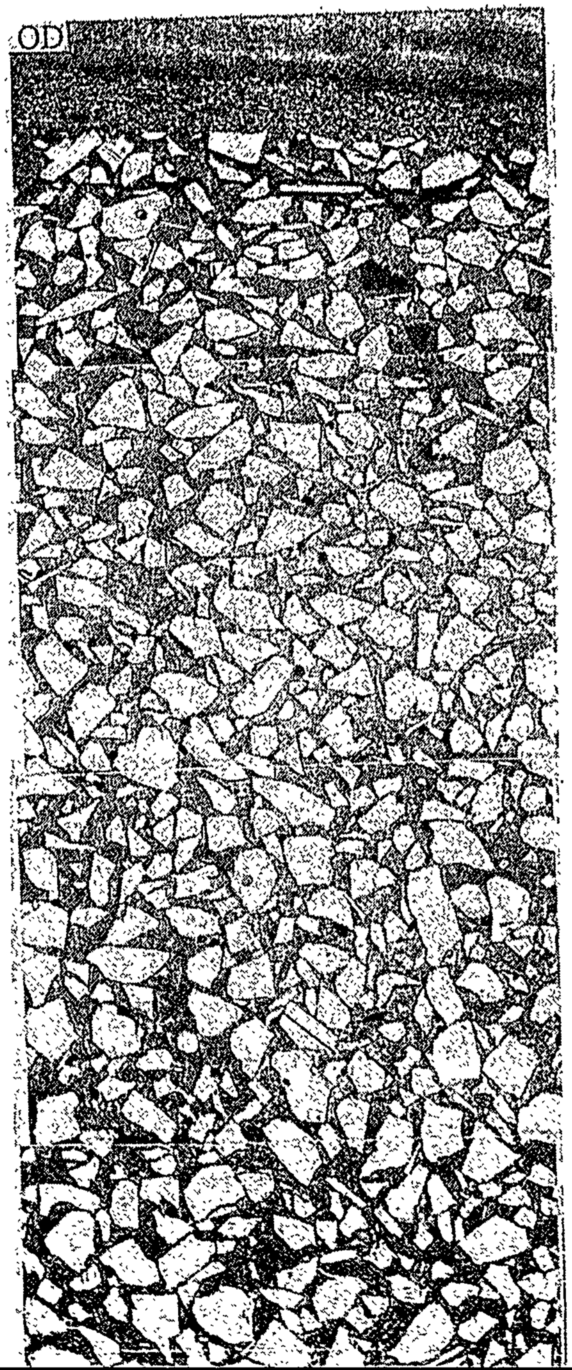


Figure 8 — Dia Schumalith 10/20 Matrix After Exposure To 10,000 Accelerated Pulse Cycles (Absence Of Fail-Safe/Regenerator)

S67/342D



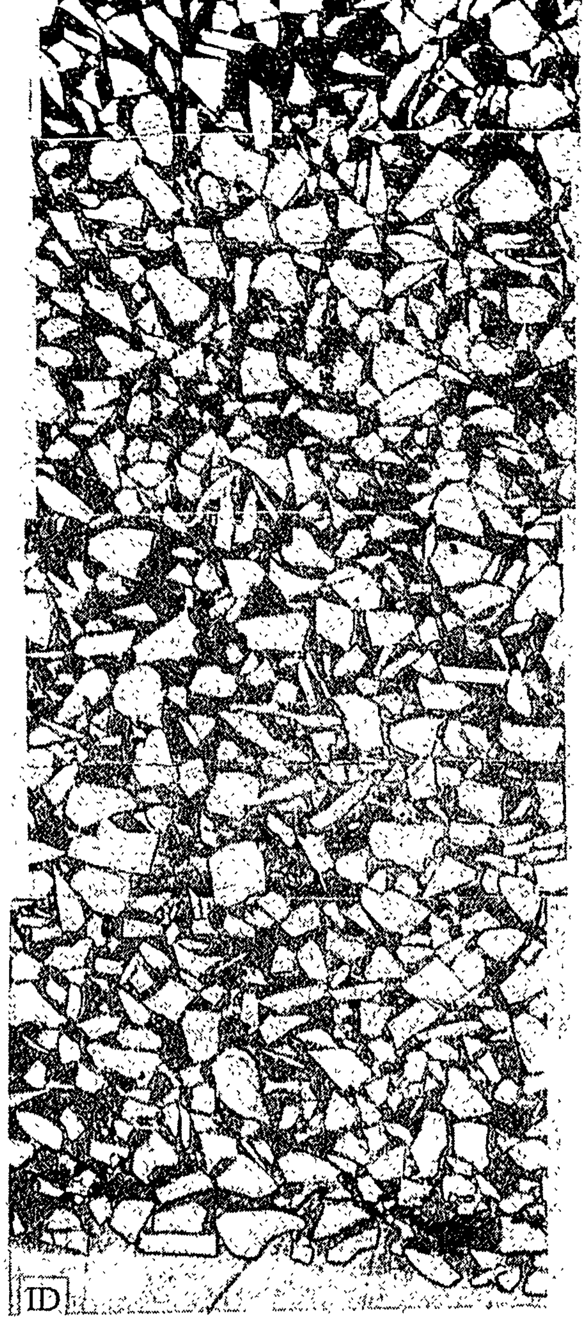


Figure 9 — Dia Schumalith 10/20 Matrix After Exposure To 10,000 Accelerated Pulse Cycles (Fail-Safe/Regenerator Included During Testing)

Crack formations were not identified along the ID wall of the Schumacher Dia Schumalith F40 filter matrix which had been exposed to comparable HTHP test conditions (Figures 10 and 11). This implies that the initially higher strength Schumacher 10/20 filter matrix may possibly respond to thermal fatigue in a similar fashion as that demonstrated by the brittle, porous, oxide-based, ceramic filter materials.(1)

S2099/352C (Top)

S2099/352C (Center)

S2099/352C (Bottom)

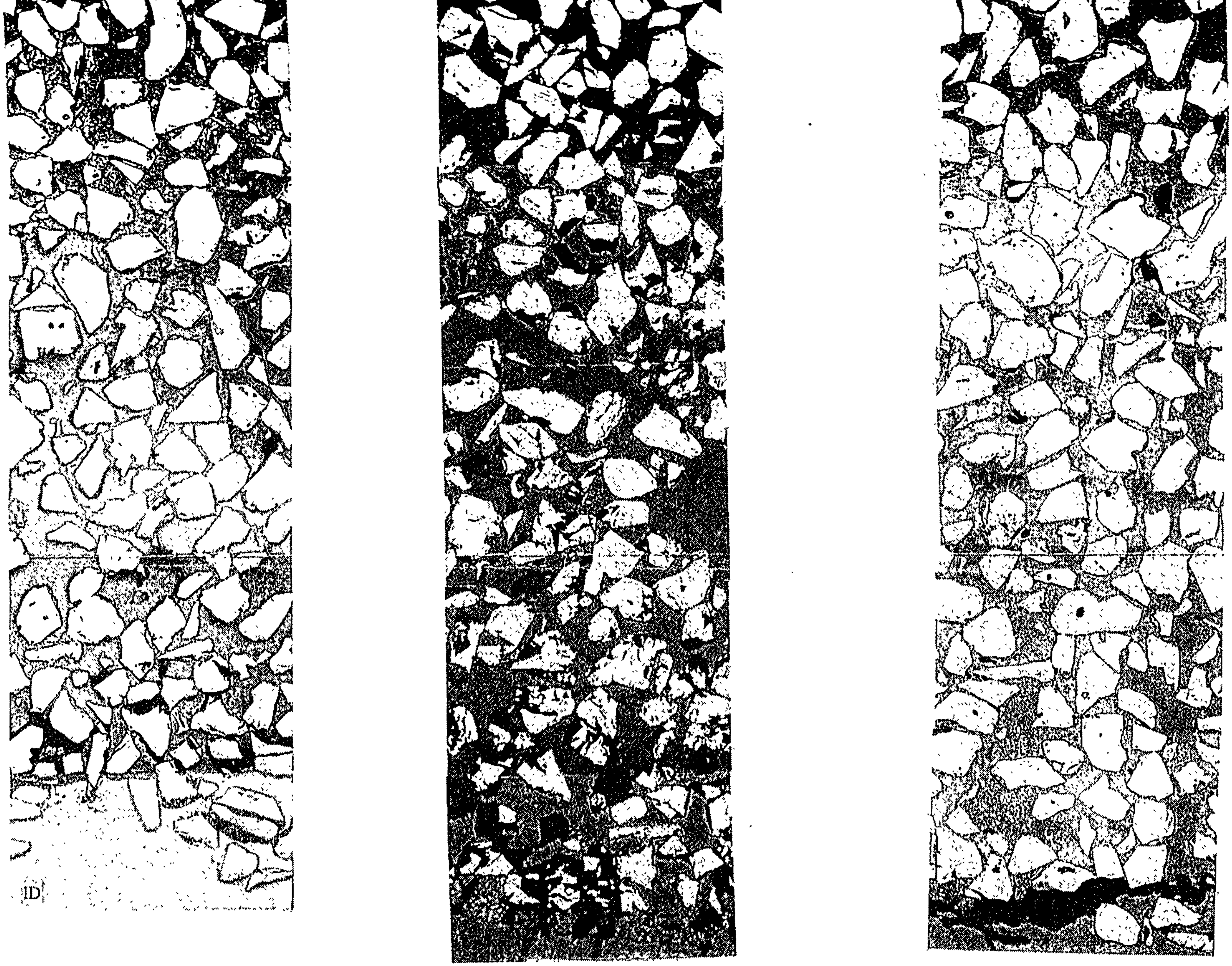


Figure 10 — Dia Schumalith F40 Filter Matrix After Exposure To 10,000 Accelerated Pulse Cycles (Absence Of Fail-Safe/Regenerator)

S2103/352C

OD

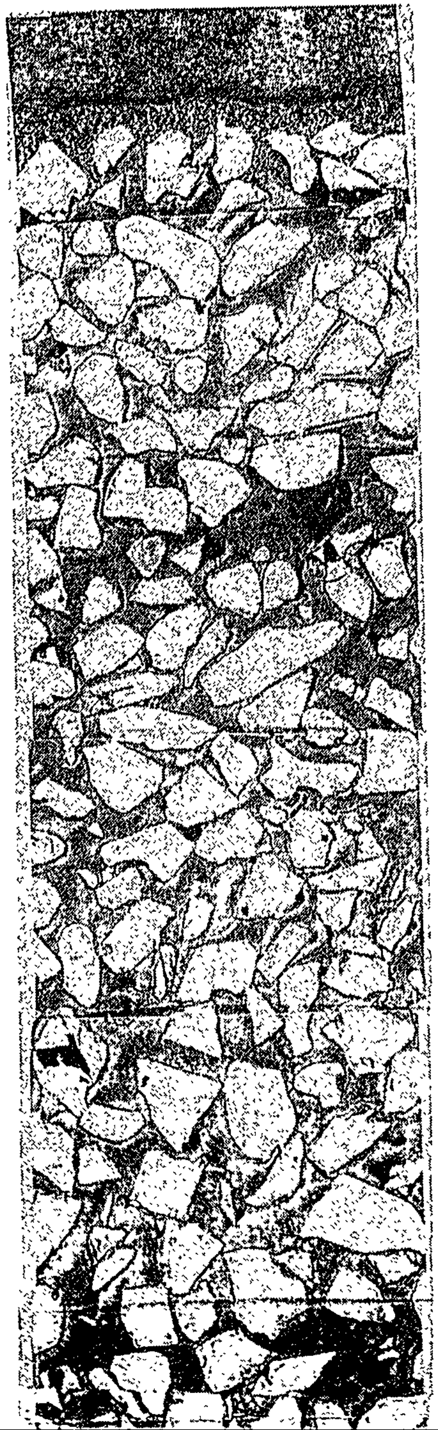


Figure 11 — Dia Schumalith F40 Filter Matrix After Exposure To 10,000 Accelerated Pulse Cycles (Fail-Safe/Regenerator Included During Testing)

RECOMMENDED PROCESS OPERATING TEMPERATURES

During extended field operation at high temperature, creep may limit the use of the clay bonded silicon carbide materials in PFBC and/or PCFBC applications where temperatures exceed 850°C (1560°F).[b] In order to assure long-term life (i.e., 1-3 years), operation of PFBC or PCFBC filter systems at temperatures below 850°C (1560°F) is recommended. Alternately for reducing gas applications, maximum operating temperatures of 650°C (1200°F) are recommended due to what has been considered as active oxidation of the silicon carbide grains which results in hole, pit, or void formations in the binder phase. Depending on the oxygen partial pressure within the reducing gas environment, passive oxidation of the silicon carbide grain may also result (i.e., formation of silica and subsequent volume expansion).

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[b] *Improved creep resistant binder materials.*

SUMMARY AND CONCLUSIONS

- The as-manufactured process temperature strengths and high temperature, creep resistant properties have been improved in both commercially available clay bonded silicon carbide Schumacher and Pall candle filter product lines.

- Based on bench-scale and field test experience, Westinghouse recommends the use of the advanced clay bonded silicon carbide candle filters in high temperature PFBC or PCFBC pilot plant or demonstration test facilities. Successful achievement of extended service life (i.e., >1-3 years) of the advanced clay bonded silicon carbide filters in the 850-900 °C (1560-1650 °F) combustion gas environment, however, remains to be demonstrated.

- Although gasification applications typically operate at lower temperatures in comparison to combustion systems, the reducing gas environment may impact the stability and life of the clay bonded silicon filter matrix. Successful, long-term operation (i.e., 1-3 years) of either the original or advanced clay bonded silicon carbide filters, particularly at process temperatures which approach 600-650 °C (1110-1200 °F), also remains to be demonstrated.

REFERENCES

1. M. A. Alvin, R. E. Tressler, T. E. Lippert, E. S. Diaz, and E. E. Smeltzer, "Durability Of Ceramic Filters," Paper presented at the DOE/METC Coal-Fired Power Systems 94 – Advances In IGCC and PFBC Review Meeting, Morgantown, WV, June 21-23, 1994.

2. M. A. Alvin, T. E. Lippert, E. S. Diaz, and E. E. Smeltzer, "Thermal And Chemical Stability Of Ceramic Candle Filters," Paper presented at the DOE/METC Advanced Coal-Fired Power Systems '95 Review Meeting, Morgantown, WV, June 27-29, 1995.

3. M. A. Alvin, R. E. Tressler, T. E. Lippert, E. S. Diaz, and E. E. Smeltzer, "Thermal/Chemical Degradation Of Ceramic Cross Flow Filter Materials," Westinghouse Science and Technology Center, Work Performed under DOE/METC Contract: DE-AC21-88MC25034, May 1994 Monthly Report.

APPENDIX A

SCHUMACHER CANDLE FILTER PRODUCT INFORMATION

Product Information

SCHUMACHER
Environmental and Separation Techniques

DIA-SCHUMALITH

Ceramic Barrier Filter with a Fine Surface Membrane of mixed Grains and Fibres

1. General Material Description

DIA-SCHUMALITH consists of a porous support body of ceramically bonded silicon carbide (SCHUMALITH) which is coated with a grey fine filtering membrane, type F. This membrane, which is composed of ceramically bonded alumina fibres and fine silicon carbide granules, is sintered onto the support body.

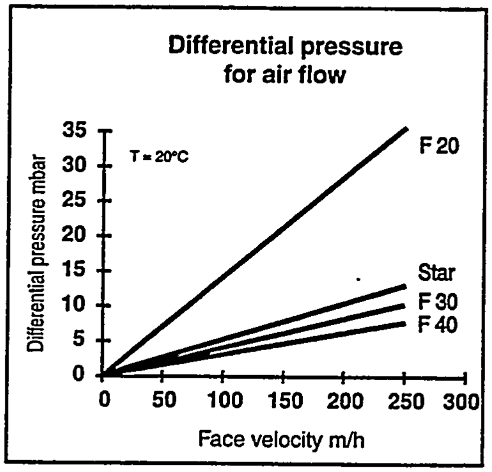
Scanning Electron Micrograph

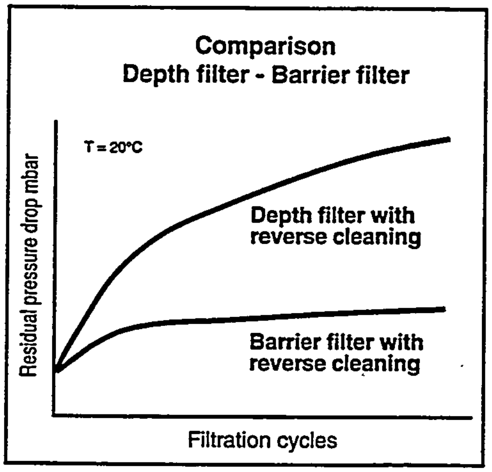
2. Physical Properties

DIA-SCHUMALITH (DSL)	Unit	Type			
		F 20*	F 30	F 40	Star
Mean Pore Size	µm	15	15	15	15
Porosity	%	36	36	37	36
Material Density	g/cm³	1,90	1.9	1.85	1.9
Specific Permeability	nPm	78	125	250	-
Pressure Drop at v = 250 m/h	mbar	35	10	7.5	13
Bending Strength	MPa	>25[1]	>20[1]	>15[1]	10[2]
Bursting Pressure	bar	-	>50	>70	>40
Max. Temperature Resistance	°C	1000	1000	1000	1000
Expansion Coefficient (25 - 1000°C)	10^{-6}/K	5.0	5.2	5.2	5.2
Thermal Conductivity (200°C)	W/m K	2.5	2.5	2.5	2.5
Dimensions do/di	mm	50/20	60/40	60/30	60/30

[1] : O-ring-strength [2] : C-ring-strength *: Custom made product
Note: Data are valid for elements of stated standard dimensions only.

3. Pressure Drop Diagrams





4. Chemical Resistance

DIA-SCHUMALITH is resistant against acids, saline solutions and organic solvents, liquid or gaseous. It does not resist hydrofluoric acid. DIA-SCHUMALITH is resistant up to pH 10 in the alkaline range.

5. Standard Dimensions

Filtercandles			
Type	do/di (mm)	l (mm)	A (m^2)
F 30	60/40	1000; 1500	0.16; 0.26
F 40	60/30	1000; 1500	0.16; 0.26
Star	60/30	1000; 1500	0.27; 0.45

Note: Special dimensions, special products and tolerances - on request.

6. Applications

DIA-SCHUMALITH is used for surface filtration. It can be cleaned on-line by reverse pulse, has a long lifetime and an excellent thermal shock resistance.

- Dust removal from hot gases and reduction of air pollution in modern power plants:
  e.g. pressurized fluidized bed combustion (PFBC) and coal gasification (IGCC)
- Cleaning of process gases and off-gases:
  e.g. biomass gasification, gasification of fossile fuels
  e.g. incineration of low-level radioactive waste
  e.g. in gas analyzing devices
- Separation and recovery of valuable substances:
  e.g. catalyst recovery
  e.g. ceramic raw products

7. General Instructions

- Elements can be glued using commercial or special ceramic glues.
  Pay attention to operating temperature and chemical resistance.

8. SCHUMACHER offers the following services:

- Filtration tests as a basis for industrial application
- Permeability tests, dust examinations
- Resistance tests
- Advice as to applications

Please apply to our expert staff for any further questions.

SCHUMACHER Umwelt- und Trenntechnik GmbH • Zur Flügelau 70 • D 74564 Crailsheim
Telefon: 0 79 51 / 30 20 • Telefax: 0 79 51 / 2 65 90 • Postfach 15 62 • D 74555 Crailsheim

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Product Information

SCHUMACHER
Environmental and Separation Techniques

DIA-SCHUMALITH

Ceramic Barrier Filter with a fine Membrane composed of Grains

1. General Material Description

DIA-SCHUMALITH is characterized by a support body of ceramically bonded silicon carbide (SCHUMALITH 20) which is coated with a white fine filtering two-layer membrane of defined pore size. This membrane, consisting of ceramically bonded mullite grains of defined particle size, is sintered onto the support body.

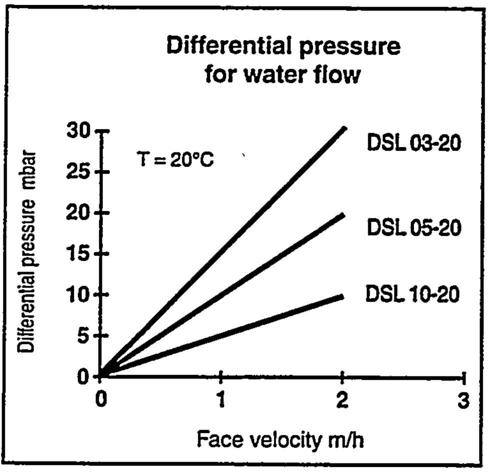
Scanning Electron Micrograph

2. Physical Properties

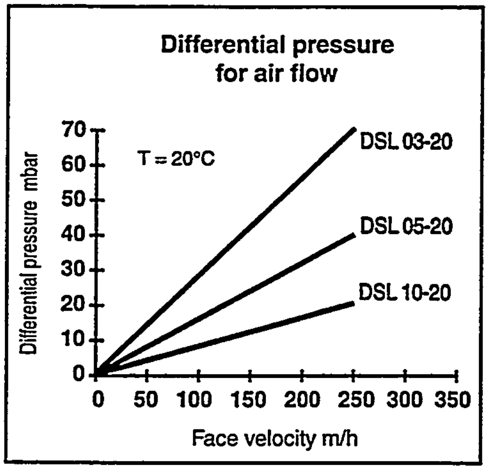
DIA-SCHUMALITH (DSL)	Unit	Type 03-20	 05-20	 10-20
Mean Pore Size	µm	3	5	10
Porosity	%	37	37	37
Material Density	g/cm³	1.9	1.9	1.9
Specific Permeability	nPm	18	30	63
Bending Strength (0-Ring-Strength)	MPa	>25	>25	>25
Bursting Pressure	bar	>50	>50	>50
Max. Temperature Resistance	°C	1000	1000	1000
Expansion Coefficient (25 - 1000°C)	10^{-6}/K	5.0	5.0	5.0
Thermal Conductivity (200°C)	W/m K	2.5	2.5	2.5
Dimensions do/di	mm	60/40	60/40	60/40

Note: Data are valid for cylinders of stated standard dimensions only.

3. Pressure Drop Diagrams



Differential pressure for water flow



Differential pressure for air flow

4. Chemical Resistance

DIA-SCHUMALITH is resistant against acids, saline solutions and organic solvents, liquid or gaseous. It does not resist hydrofluoric acid. DIA-SCHUMALITH is resistant up to pH 10 in the alkaline range.

5. Standard Dimensions

Filter cylinders			
Type	do / di (mm)	l (mm)	A (m^2)
03-20	60/40	1000	0.188
05-20	60/40	1000	0.188
10-20	60/40	1000	0.188

Note: Special dimensions, special products and tolerances - on request.

6. Applications

DIA-SCHUMALITH is used for surface filtration. It can be cleaned on-line by reverse pulse, has a long lifetime and an excellent thermal shock resistance.

- Catalyst recovery from liquids:
  e.g. in H_2O_2 -production
  e.g. in Caprolactam production
- Separation of superfine dusts:
  e.g. in fluid-catalytic cracking (FCC) processes
  e.g. in power generation processes
- Separation of abrasive dusts:
  e.g. ceramic raw products

7. General Instructions

- Special care has to be paid to the sealing when installing the filter elements.
- The filter elements should not be cut to any other length as otherwise the membrane may get damaged.
- Elements can be glued using commercial or special ceramic glues.
  Pay attention to operating temperature and chemical resistance.

8. SCHUMACHER offers the following services:

- Filtration tests as a basis for industrial application
- Permeability tests, dust examinations
- Resistance tests
- Advice as to applications

Please apply to our expert staff for any further questions.

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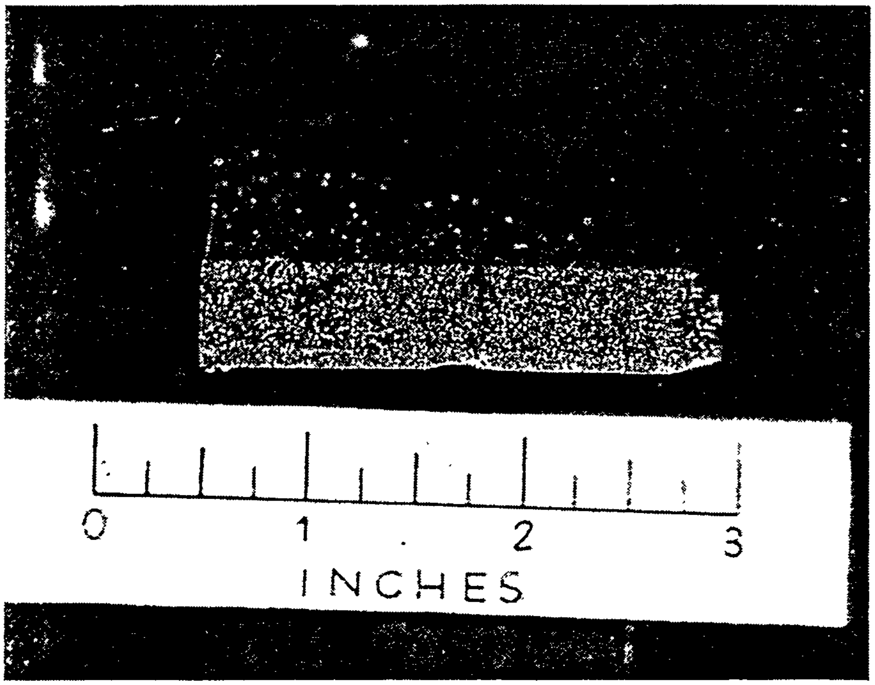
APPENDIX B

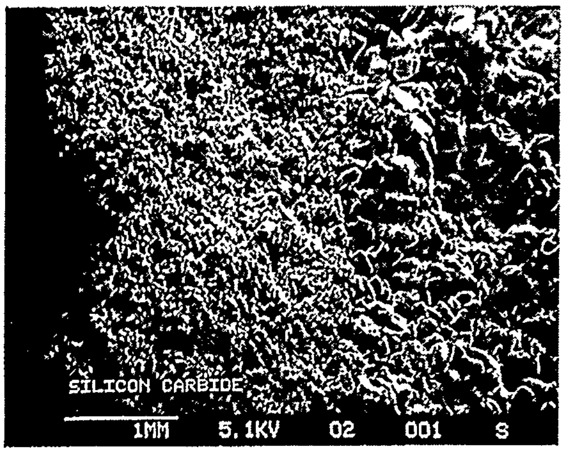
REFRACTRON/PALL CANDLE FILTERS
-- FILTER MATERIALS DEVELOPMENT --

Refractron SiC

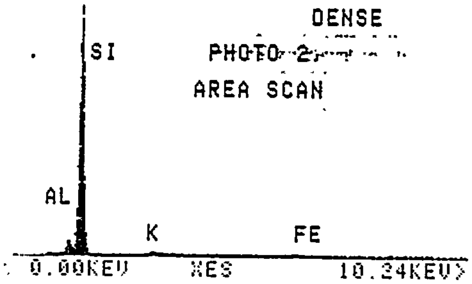


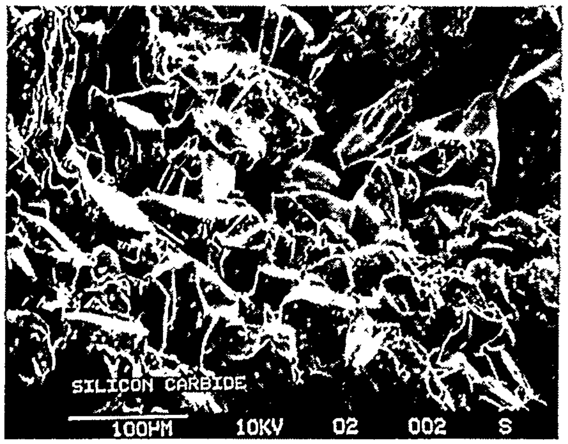
The Refractron filter material consists of a double layer, crystallized glass bond SiC matrix. The dense outer layer of the Refractron material is approximately 2-2.5 μm thick, and consists of fine silicon carbide grains (80 μm). The dense outer layer is supported by a coarse porous inner matrix of 200 μm SiC grains.

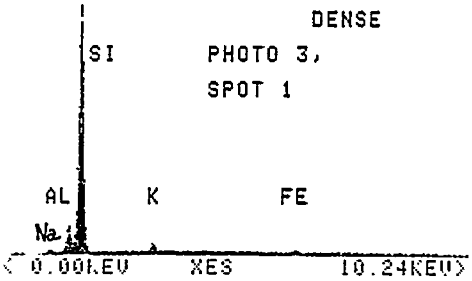
Refractron SiC

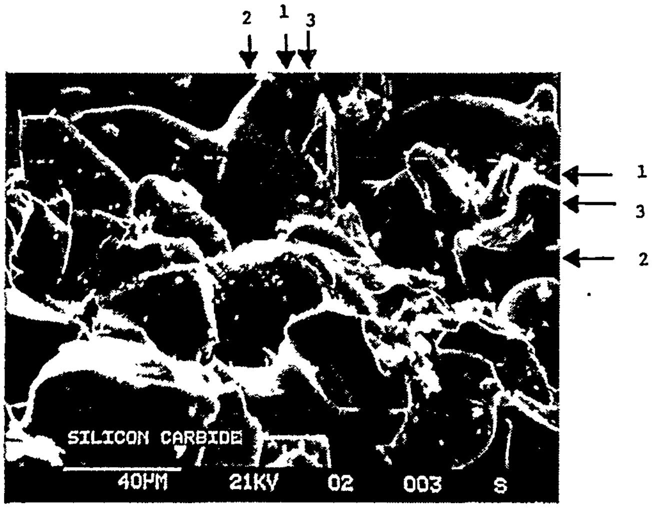


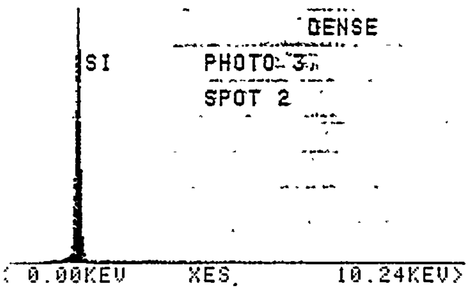
Micrograph illustrates the morphology of the fractured, cross-sectioned, Refractron SiC surface. A compositional analysis area scan of this surface indicates that the material consists of silicon, aluminum, potassium, and iron.

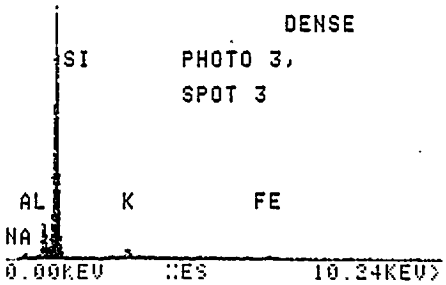








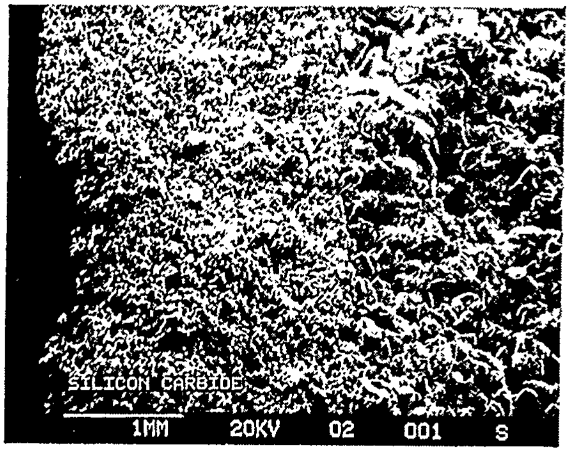




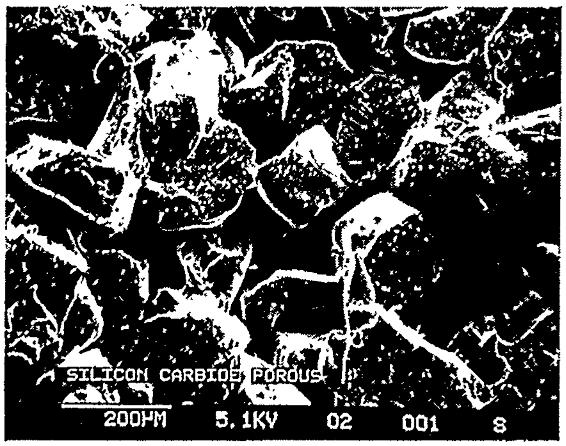
Area 1 and Area 3 illustrate the morphology of the crystallized glass bond which envelopes the underlying SiC grains. The matrix consists of aluminum, sodium, potassium, and iron, and possibly silicon. Since the underlying matrix is SiC, the contribution of silicon within the glass bond is not determined due to the uncertainty in defining the thickness of the glass bond layer. This phase is considered to be the preferential site for reaction with gas phase alkali at high process temperature, as well as a location for fines attachment.

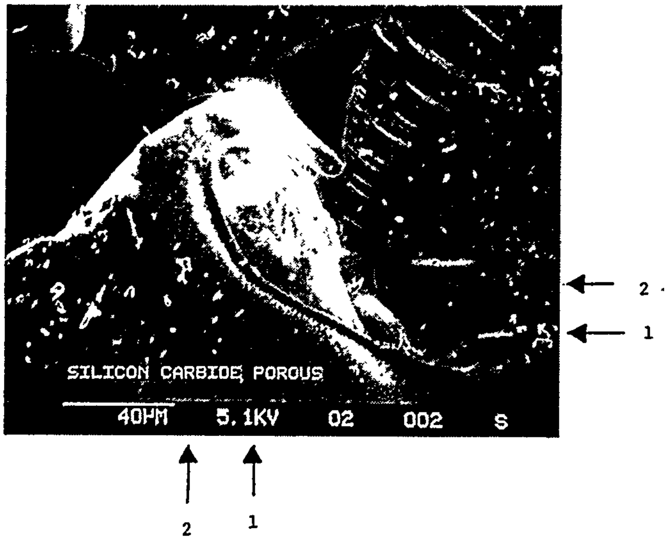
Area 2 shows the fracture surface of a SiC grain. Only silicon and not carbon in the SiC structure can be identified by energy dispersive x-ray analysis (EDAX).

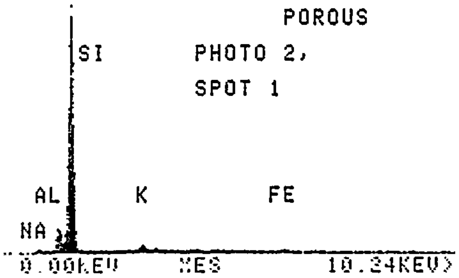
Refractron SiC

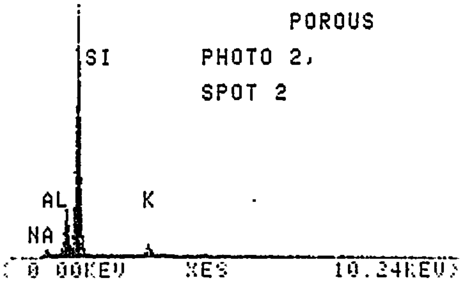


Higher magnification micrograph illustrating the coarse SiC grains.



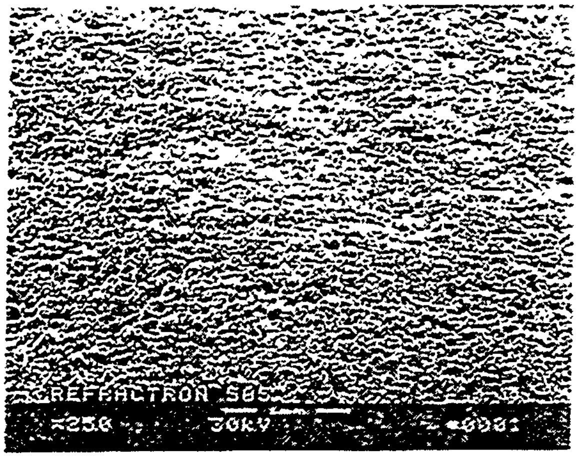




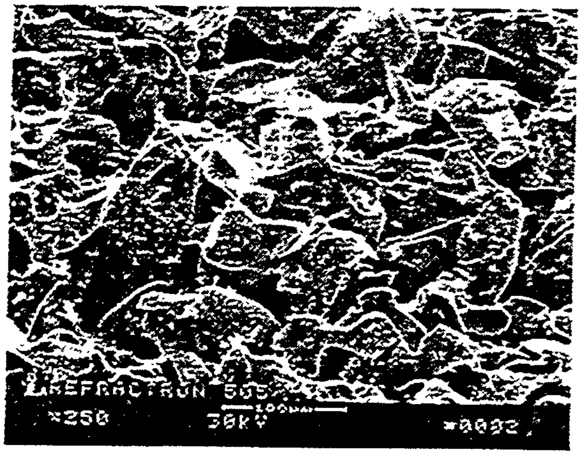


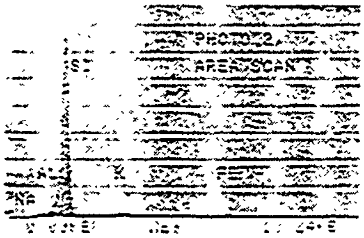
Higher magnification micrograph illustrating the grain-to-grain interconnecting phase. Note that the SiC grains are enveloped by an aluminum, and possibly silicon, sodium, potassium, and iron-rich binder-like phase. Also note the crack which has formed along the interface material, at the base of adjoining grains.

Refractron SiC (505)



The microstructure of the Refractron SiC (505) matrix is shown in this micrograph. This material contains a modified binder phase in comparison to the traditional Refractron SiC (501) matrix.

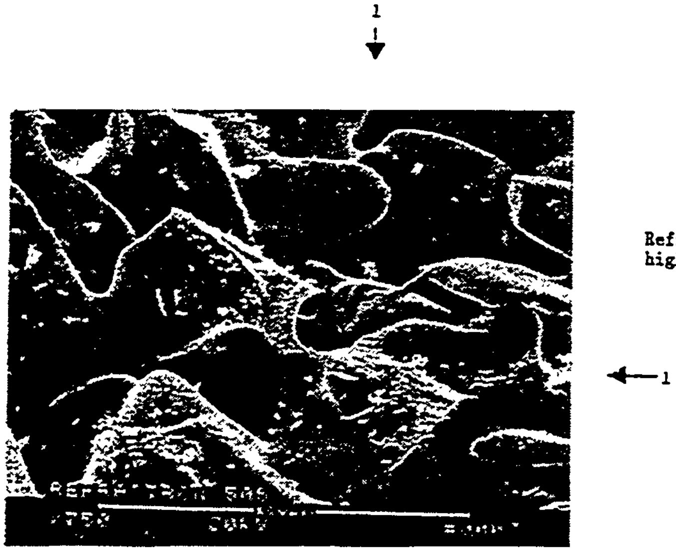




This higher magnification micrograph shows the presence of coarse SiC grains which appear to be held together with a binder-like matrix. EDAX area scan analysis shows:

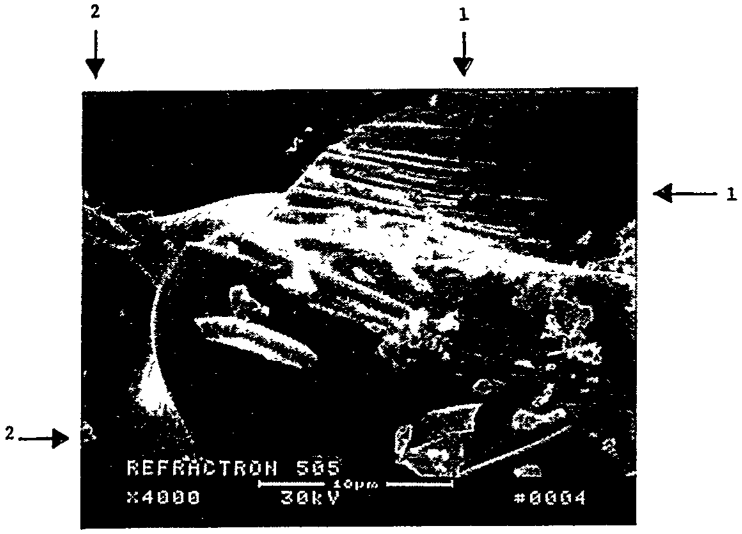
Atomic percent distribution
Normalization factor = 0.902

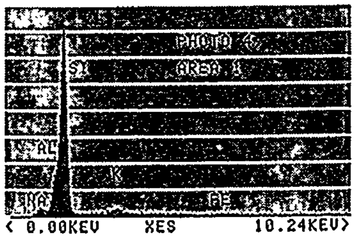
6.27% Al; 91.22% Si; 1.08% K; 0.68% Fe

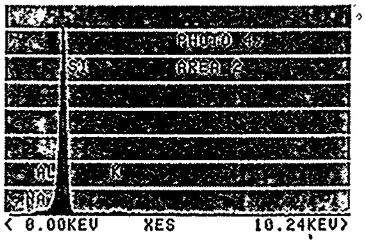


Higher magnification of the fractured Refractron SiC (505) surface. Area 1 is shown at higher magnification in the following micrograph.

Refractron SiC (505)

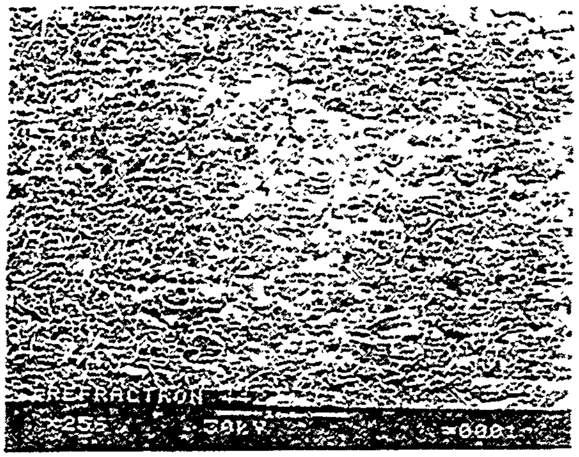


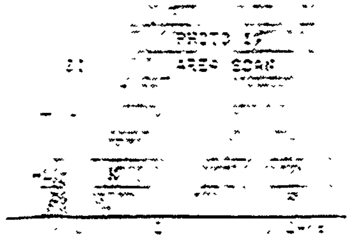




Area 1 along the fracture surface consists of Na, Al, Si, K, and Fe, implying the presence of a binder phase. Area 2 contains a somewhat lower Na, Al, K, and Fe content, perhaps implying a thinner layer of the binder along the SiC grain.

Refractron SiC (442)

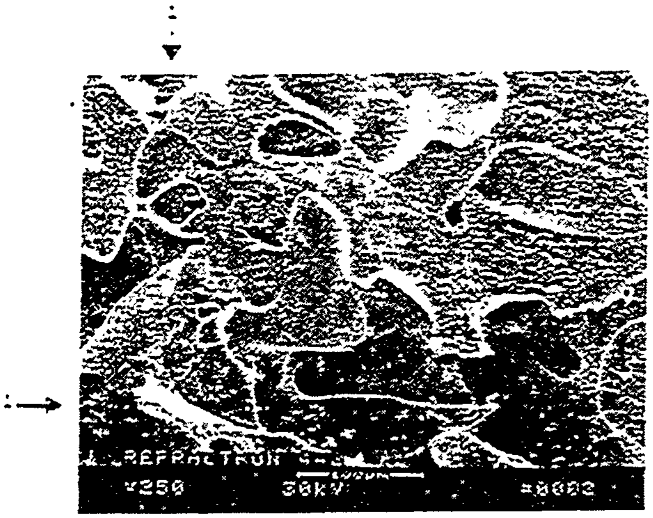




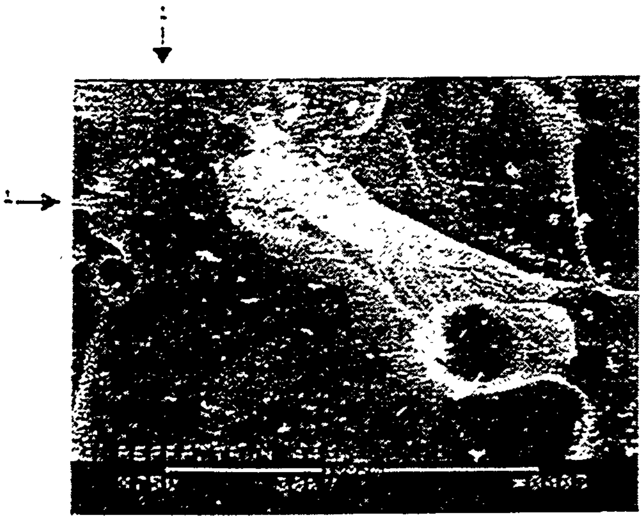
This micrograph illustrates the morphology of the modified Refractron SiC (442) matrix. In comparison with Refractron SiC (505) this material appears to contain larger grains, and a more open porosity. An EDAX area scan indicates an

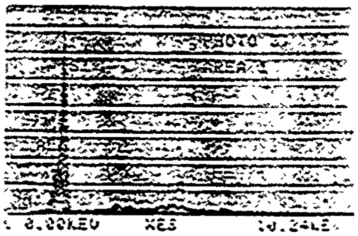
Atomic percent distribution
Normalization factor = 0.914

6.50% Al; 91.75% Si; 1.74% K



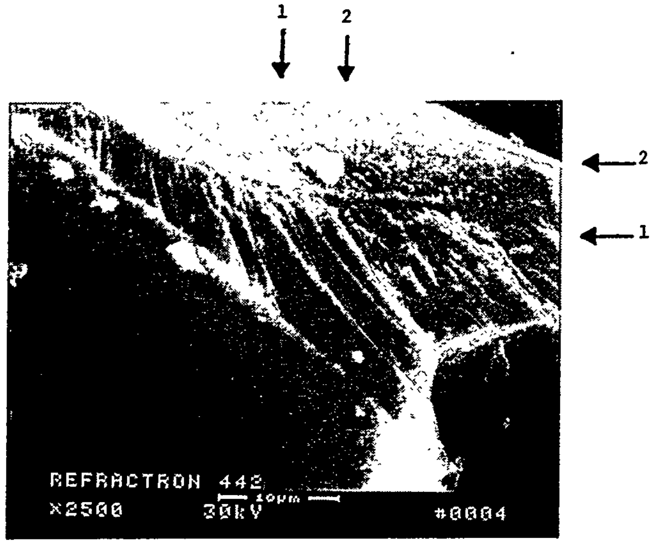
At higher magnification, the fractured surface does not appear to contain discrete SiC grains, but a more non-descript binder-coated matrix. In comparison with the Refractron SiC (505), the 442 matrix appears to contain a higher binder content (6.50 vs 6.27% Al; 1.74 vs 1.03% K; 442 vs 505, respectively). Notice the quantity of "fines" which are present along this surface. A higher magnification micrograph of Area 1 is shown below.

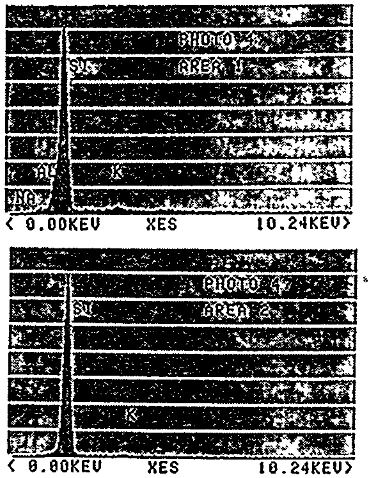


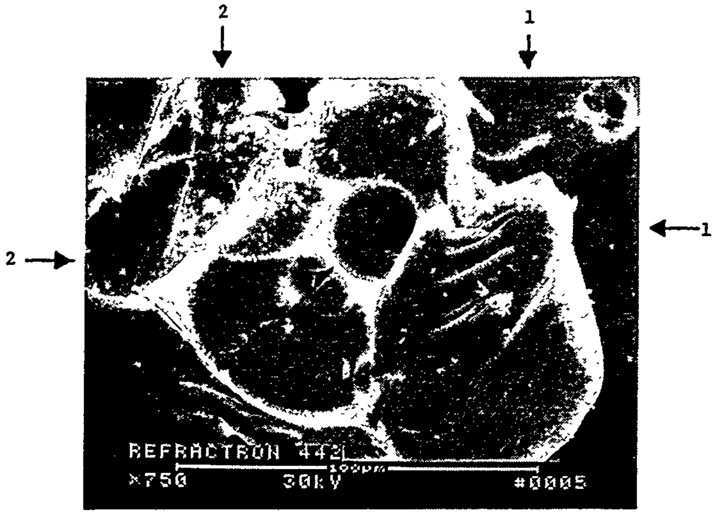


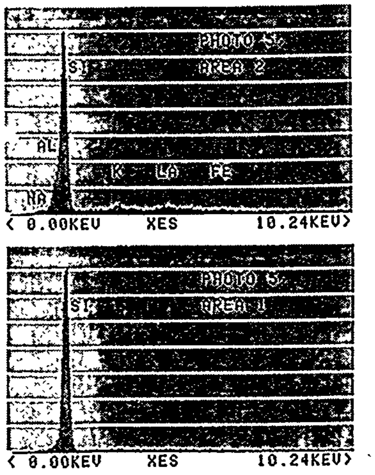
Higher magnification micrograph attempting to illustrate a fracture along the SiC grain-binder interface. Notice the bubble-like formation in the binder phase. Analysis of Area 1 indicates the presence of Al, (Si), K, La, and Fe in the binder interconnect phase. Higher magnification of the grain-binder fracture is shown in the following micrograph.

Refractron SiC (442)









This micrograph illustrates the morphology of the Refractron SiC 442 matrix at an alternate location. Area 1 reflects the composition of the SiC grain, while Area 2 appears to contain the binder phase (Na, Al, (Si), K, La, and Fe). Notice the bubble formations in the binder interconnect material, and separation or cracking at the binder-grain interface.