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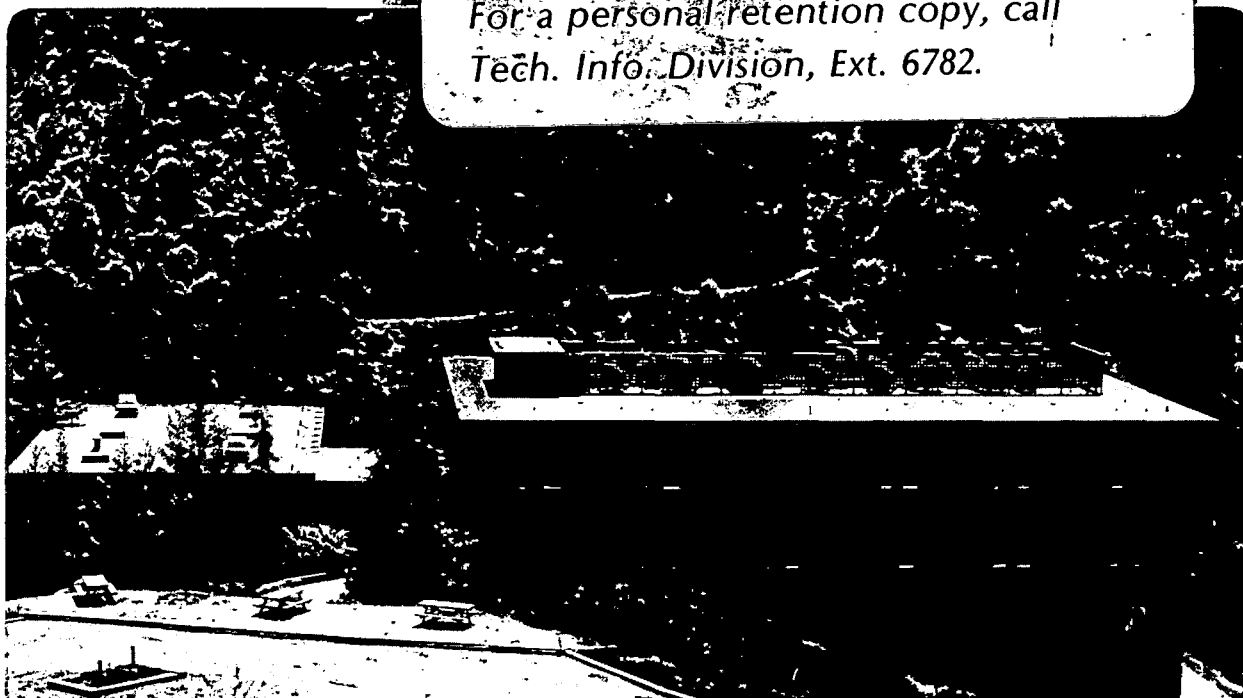
FORMATION OF MULLITE IN KAOLINITE AND  $\text{Al}_2\text{O}_3\text{-SiO}_2$   
MIXTURES

Sylvia M. Johnson and Joseph A. Pask

July 1982

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# FORMATION OF MULLITE IN KAOLINITE

## AND $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$ MIXTURES

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### ABSTRACT

Effect of addition of impurities present in kaolinite -  $\text{TiO}_2$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$  - to equivalent  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixtures on crystal size and morphology of mullite were studied at 1650-1700°C.  $\text{CaO}$  and larger amounts of  $\text{Fe}_2\text{O}_3$  added to  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixtures and to kaolinite caused an increase in crystal size. Kaolinite-based mixtures always showed acicular crystals which were observed in an  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixture only with larger additions of  $\text{Fe}_2\text{O}_3$ .

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## 1. INTRODUCTION

A comparison of the formation of mullite from kaolinite and an equivalent alumina-silica mixture and the effect of additives on microstructure development at high temperatures have not been studied adequately. A relatively pure Georgia kaolinite whose main impurities were  $\text{TiO}_2$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Na}_2\text{O}$  and  $\text{K}_2\text{O}$  was chosen for this study. Each impurity was added to an  $\text{Al}_2\text{O}_3$  mixture in proportions similar to those in the kaolinite. Calcium oxide and iron oxide, in equivalent molar amounts were also added to the  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixture and the kaolinite. Scanning electron microscopy was used to study the microstructures of samples subjected to several heating schedules in the temperature range  $1650^\circ$ - $1700^\circ\text{C}$ .

Numerous researchers have investigated the effect of additives on mullite formation in kaolinite, generally at temperatures up to  $1400^\circ\text{C}$  in air. Those oxides most commonly studied were  $\text{Fe}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{CaO}$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{MgO}$ , and alkali oxides. The amount of mullite formed, physical properties and microstructure have been investigated.

Iron oxide is one of the most commonly found impurities in kaolinite.<sup>1</sup> Its effect on kaolinite,<sup>2-5</sup> alumina-silica mixtures,<sup>6,7</sup> and mullite<sup>1</sup> has been investigated.  $\text{Fe}_2\text{O}_3$  forms a solid solution with mullite.<sup>1,5,8</sup> Brownell<sup>1</sup> found a limit of 7.7% in solid solution with mullite at  $1300^\circ\text{C}$ . Muan<sup>6</sup> observed that 10%  $\text{Fe}_2\text{O}_3$  will substitute for  $\text{Al}_2\text{O}_3$  in mullite formed from kaolinite at  $1370^\circ\text{C}$  while Gelsdorf et al<sup>9</sup> found that 6% would enter into the mullite structure at  $1300^\circ\text{C}$  and 8-9% at  $1400^\circ\text{C}$ . Murthy and Hummel<sup>10</sup> concluded that the limit was 10-12% at  $1300^\circ\text{C}$ , agreeing with the evidence of Agrell and Smith<sup>11</sup> and McGee and Wirkus<sup>12</sup> who concluded that 12% was the limit at  $1300^\circ\text{C}$  for alumina-silica mixtures.  $\text{Fe}^{3+}$

substitutes for  $Al^{3+}$  in mullite as a result of its size and ionic charge<sup>3,8,10,13</sup>. Budnikov<sup>14</sup> has reported the formation of needles of mullite when mullite is in contact with glass containing iron oxide. McGee and Wirkus<sup>12</sup> reported that the mullite crystals originating from  $Al_2O_3-SiO_2$  were well formed when  $Fe_2O_3$  was present and Chaudhuri<sup>5</sup> found that  $Fe_2O_3$  enhanced growth of mullite formed from kaolinite.

Titania is also a commonly occurring impurity in kaolinite. The amount which enters into solid solution with mullite is much less than that of  $Fe_2O_3$ .<sup>2,5,10,12</sup> Gelsdorf et al.<sup>9</sup> report that 2-2.5%  $TiO_2$  is included in mullite formed from  $Al_2O_3-SiO_2$  mixtures after firing at 1700°C for eight hours while McGee and Wirkus<sup>12</sup> report that the limit is 3%. Murthy and Hummel<sup>10</sup> have found that the amount of  $TiO_2$  entering into the mullite structure is 2-4% which is in agreement with the results of Agrell and Smith.<sup>11</sup>

Calcium effect on mullite formation has been studied. Chaudhuri<sup>5</sup> found that CaO added to kaolinite produced much larger crystals than other additives.  $Na_2O$  and  $K_2O$  were shown to be poor mineralizers and hindered the growth of mullite from kaolinite.<sup>2,5,13,15</sup>

## 2. MATERIALS AND PROCEDURE

Materials used were  $\alpha$ -alumina A-14<sup>\*</sup>, silica flour<sup>\*\*</sup> ( $\alpha$ -quartz) and a relatively pure Georgia Kaolin<sup>\*\*\*</sup>. Chemical analyses of these materials are given in Table 1. The oxides and sodium carbonate were of analytical

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\* Alcoa Aluminum Corporation of America.

\*\* Ottawa Silica Co.

\*\*\* Georgia Kaolin Co.

reagent grade. Compositions of the mixtures that were prepared are given in Table 2. The basis of the  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixtures is the composition of the kaolinite. Mixtures VII to X contain more  $\text{CaO}$  and  $\text{Fe}_2\text{O}_3$ ; the molar amount of oxide added in each of these cases was the same.

The oxides were mixed and ground in a vibrating mill<sup>\*</sup> with alumina grinding media and isopropyl alcohol for ~16 hours. The slurry was dried and crushed with a mortar and pestle. The powders were sprayed lightly with water and mixed on a roller mill for a few minutes to form a free flowing, granular powder. They were compacted in a 9 mm diameter steel die under a uniaxial pressure of ~50 MPa. The pellets were prefired<sup>\*\*</sup> to 1000°C to improve their strength. The kaolinite was prefired to 1200°C, crushed and made into pellets.

After prefiring, the samples were fired on a platinum disc in a vertical quench furnace that was electrically heated with  $\text{MoSi}_2$  elements. Firing schedules consisted of 8 and 24 hours at 1650°C, 8 and 24 hours at 1700°C, and 8 hours at 1700°C followed by 16 hours at 1650°C. The samples were taken up to temperature as quickly as possible, as was reduction in temperature from 1700°C to 1650°C. The fluctuation at temperature was  $\pm 5^\circ\text{C}$ . At the end of the run, the samples were quenched by lowering the pedestal.

Pellets were cut on an Isomet saw and mounted in bakelite. Polishing was on 30 $\mu\text{m}$ , 15 $\mu\text{m}$  and 6 $\mu\text{m}$  diamond wheels followed by 6 $\mu\text{m}$ , 1 $\mu\text{m}$  and 0.25 $\mu\text{m}$  Syntrons. The samples were etched in 40% HF for ~30 seconds prior to

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\* Sweco Inc., Los Angeles, CA.

\*\* Lindberg, Watertown, WI.

SEM examination at 1000 and 2000 X magnification.

### 3. RESULTS AND DISCUSSION

#### A. Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> Mixture

Time and temperature effects on heating the Al<sub>2</sub>O<sub>3</sub> mixture are shown in Fig. 1. The stable equilibrium phases at 1650-1700°C are mullite and an alumino-silicate liquid; on quenching, if sufficiently rapid, the liquid solidifies to form a glass. The mullite forms on the surfaces of the Al<sub>2</sub>O<sub>3</sub> grains and grows by interdiffusion of Al<sup>3+</sup> and Si<sup>4+</sup>. Dissolution of mullite into the liquid occurs simultaneously until the liquid is saturated with mullite. The shape of the original mullite crystals is determined by the Al<sub>2</sub>O<sub>3</sub> particles and is essentially as seen in Fig. 1A which shows the appearance after firing at 1650°C for 8 hours. The crystals are small, rounded and tend to be agglomerated, which reflects the original aggregation of the starting alumina powder.

After phase equilibrium is reached, mullite crystal growth continues by Ostwald ripening, a process of solution of small particles and precipitation onto larger crystals. This process is thermodynamically favorable because of the resulting decrease in surface area and therefore total surface energy. The specific driving force is the higher energy and solubility (according to Kelvin's equation) of the smaller particles resulting in supersaturation of the liquid relative to the larger particles. Rate of growth is affected by the rate at which the ions or molecules diffuse in the liquid, this in turn being affected by temperature, viscosity and liquid composition (i.e. the presence of impurities).

Figure 1B shows a sample heated at 1650°C for 24 hours; the crystals appear larger and more separated in comparison with those heated for 8



hours (Fig. 1A). The shape has also changed and the crystals have elongated and formed flat faces although the ends still tend to be rounded. Elongation or change in shape is the result of differences in surface energies of the different crystal planes. A low value of  $\gamma_{sl}$ , the interfacial solid-liquid energy for a particular crystallographic face, causes the growth of that face with the development of flattening.

Comparison of Figs. 1C and 1D with Figs. 1A and 1B shows the effect of a 50°C rise in temperature. The crystals are much larger, more distinct and well formed. Diffusivities are greater at higher temperatures and equilibrium phase compositions are reached much more rapidly. Examination indicates that the relative size increase between 8 and 24 hours, is not as large at 1700°C as it is at 1650°C. The faster relative growth rate at the lower temperature can be explained on the basis of a non-linear size vs. time curve which approaches a steady state value.

A curve of the form of Fig. 2 with a different time scale for each temperature is such an example. At 1700°C the most rapid growth occurs before 8 hours and is approaching the steady value at 8 hours. Therefore, the change in size between 8 and 24 hours corresponds only to the distance AB on the y axis. At 1650°C, growth is rapid between 8 and 24 hours, resulting in an increase in size equivalent to CD. Since growth by Ostwald ripening starts after phase equilibrium is reached, another contributing factor to the differences in growth rate is that growth at 1650°C is comparatively more delayed.

Figure 1E is the result of the two stage firing process with a 1700°C hold for 8 hours, followed by 16 hours at 1650°C. The crystals have the same shape as those for the 1700°C firings but are smaller in size than

those fired at 1700°C for 8 hours (comparison of Figs. 1E with 1C). It could be expected that the crystals in Fig. 1E would be larger since the sample was fired for an additional 16 hours at 1650°C and the equilibrium amount of mullite is greater at 1650°C than at 1700°C. This phenomenon could be explained as follows. With phase equilibrium at 1700°C, the activity of alumina,  $a_{\text{Al}_2\text{O}_3}$ , is constant in mullite and in the liquid. When the temperature is lowered to 1650°C, the value of  $a_{\text{Al}_2\text{O}_3}$  at equilibrium is less and the liquid becomes supersaturated with mullite of lower  $a_{\text{Al}_2\text{O}_3}$ . Thus mullite does not precipitate onto the mullite with the high  $a_{\text{Al}_2\text{O}_3}$  and nuclei form. The original mullite with a higher  $a_{\text{Al}_2\text{O}_3}$  goes into solution as the new mullite grows until the equilibrium  $a_{\text{Al}_2\text{O}_3}$  is reached throughout the system. The net result is a larger number of smaller crystals.

#### B. Kaolinite

Kaolinite fired under different conditions is represented in Fig. 2. The morphological difference in the mullite crystals on comparison with the  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixture, Fig. 1, is readily noticeable. The mullite crystals in Fig. 1 are rectangular in shape with rounded ends while the mullite crystals formed from kaolinite are largely acicular with pointed ends and diamond-shaped cross-sections.

The mechanisms for mullite formation are different. In mixtures of  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  mullite forms by interdiffusion through the forming mullite. Kaolinite, however, undergoes a series of reactions initiated by a dehydroxylation reaction followed by a progressive exsolution of  $\text{SiO}_2$ .<sup>16</sup> In addition, kaolinite contains significant amounts of impurities.

An  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixture was prepared with additions of impurities equivalent in amount to those present and measurable in kaolinite (Table 1). This specimen was fired at  $1700^\circ\text{C}$  for 8 hours followed by 16 hours at  $1650^\circ\text{C}$  and compared with a similarly fired kaolinite sample (Figs. 3E and 3D, respectively). The acicular crystals that were present in the kaolinite did not develop indicating that other factors play a more critical role in controlling morphology. A comparison of similarly fired mixtures of  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  without and with all of the impurities (Figs. 1E and 3E) showed an increase in crystal size but no significant change in morphology.

A comparison of Figs. 3B and 3C indicates that the principal growth of the crystals is lengthwise in the 'c'-direction suggesting that the crystal faces parallel to the 'c'-axis have lower interfacial energies. Whether this situation exists because of surface structural differences due to the starting kaolinite structure or because of differences in the distribution of the impurities is not known. Relics of small crystals can be seen in both of these photographs, indicating that the small crystals are being dissolved and that growth is by Ostwald ripening.

A comparison of Figs. 3D and 3B, shows that a small growth of the grains occurred when the specimen was heated an additional 16 hours at  $1650^\circ\text{C}$  after first heating at  $1700^\circ\text{C}$  for 8 hours in contrast to a decrease in grain size for an  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixture (Figs. 1C and 1E). Phase equilibrium was probably achieved rapidly enough on lowering the temperature to  $1650^\circ\text{C}$  to allow some growth which can be attributed to the higher impurity content of the kaolinite. Another possible factor may be a lower  $a_{\text{Al}_2\text{O}_3}$  at the higher temperature due to the formation of mullite

by exsolution of  $\text{SiO}_2$  from dehydroxylated kaolinite.

C.  $\text{Al}_2\text{O}_3$  -  $\text{SiO}_2$  Mixtures With Oxide Additions

Samples of  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixtures with separate additions of 1.4 wt%  $\text{TiO}_2$ , 0.39  $\text{Fe}_2\text{O}_3$ , and 0.38  $\text{Na}_2\text{O}/\text{K}_2\text{O}$  which were in the same proportions as in the kaolinite (Table 1) were prepared. They were fired at 1700°C for 8 hours followed by 1650°C for 16 hours. Microstructures are shown in Fig. 4. Comparisons can be made with the microstructure of a similarly fired specimen with no additive (Fig. 1E) and with all of the oxides added (Fig. 3E).

All the samples have similarly shaped grains which tend to be rectangular with varying degrees of roundness at the ends, but the grain sizes vary. The  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  sample, Fig. 1E, and that with  $\text{Na}_2\text{O}$  and  $\text{K}_2\text{O}$ , Fig. 4C, have the smallest crystals followed by the sample with  $\text{TiO}_2$ , Fig. 4A, with 0.34%  $\text{Fe}_2\text{O}_3$ , Fig. 4B, and with all of these oxides, Fig. 3E, which has larger and more blocky crystals than kaolinite, Fig. 3D. The grains become better formed, i.e. straight side prominence, with increase in grain size. The amount of mullite formed in this series does not appear to vary significantly.

Additions of 2.0 wt%  $\text{CaO}$  were also made to an  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixture (Fig. 4D), and to kaolinite (Fig. 4E) for comparison. The mullite crystal formed in the former specimen are the largest of all of the additives, equivalent in amount to that in kaolinite. The crystals are also larger in the kaolinite specimen (compared with Fig. 3D). Bloating was observed in all the  $\text{CaO}$  samples, making them very porous.

D. Additions of  $\text{Fe}_2\text{O}_3$

The effects of additions of 5.7 wt%  $\text{Fe}_2\text{O}_3$  (equivalent to molar amount of 2.0 wt%  $\text{CaO}$  added) are compared in Figs. 5A and 5B for the two-stage heat treatment, 8 hours at  $1700^\circ\text{C}$  followed by 16 hours at  $1650^\circ\text{C}$ . These specimens have very similar appearances.  $\text{Fe}_2\text{O}_3$  in the larger amounts is the only additive to the  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixture that caused the formation of acicular crystals (Fig. 5A) which were maintained in the kaolinite specimen (Fig. 5B). Similar compositions fired at  $1650^\circ\text{C}$  for 24 hours are shown in Figs. 5C and 5D, and at  $1700^\circ\text{C}$  for 24 hours, in Figs. 5E and 5F.

The persistence of the acicular shape of the mullite crystals formed from all kaolinite-based mixtures and its appearance in the  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixture containing  $\text{Fe}_2\text{O}_3$  is expected to be related to the mechanism of the respective mullite forming reactions mentioned earlier and/or to impurity induced anisotropy of interfacial energies that persist during heat treatments. Iron oxide enters into solid solution in amounts of up to 10-12% while other oxides have limited solubilities. Impurities associated with the kaolinite may be incorporated in the mullite structure during its method of formation and play a role in the development of the anisotropy of surface energies. In the case of the  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixtures the formation of mullite by interdiffusion does result in anisotropy, but not to the same extent as indicated by its morphology. An incorporation of a sufficiently large amount of  $\text{Fe}_2\text{O}_3$  in solid solution apparently does produce the degree of anisotropy of surface energies leading to the observed formation of acicular crystals.

An additional feature appears in the microstructure of Fig. 5F, and a small amount of it also in Fig. 5E. A section of Fig. 5F is shown at higher magnification in Fig. 6. The feature was too small for analysis. Its fine texture and an examination of the  $\text{FeO} \cdot \text{Fe}_2\text{O}_3$ - $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  phase diagram<sup>6</sup> suggests that it would be a two-phase mixture precipitated along the mullite-cristobalite phase boundary. This phenomenon is a point of interest because it appeared only with the larger  $\text{Fe}_2\text{O}_3$  addition primarily in the kaolinite mixture and only after heat treatment at  $1700^\circ\text{C}$  prior to cooling. The viscosity of the liquid prior to its solidification appears to be most critical. The larger amount of overall impurities would be expected to reduce the viscosity of mixtures containing kaolinite for comparable heating conditions. Presence of  $\text{Fe}_2\text{O}_3$  in the liquid would also be expected to have a greater driving force for two-phase crystallization since the ternary eutectic liquid for this system contains  $\sim 40\%$   $\text{SiO}_2$  and for the other additive systems it contains  $\sim 60$ - $70\%$   $\text{SiO}_2$ , the liquids in each case having about  $80\%$   $\text{SiO}_2$  at the start of two-phase crystallization. It can also be expected that the structure of the liquid is significantly different after heat treatment at  $1700^\circ\text{C}$  as compared with heat treatments at  $1650^\circ\text{C}$  on the basis of crystallization behaviors.

### 3. CONCLUSIONS

The formation of mullite from kaolinite and  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixtures occurs by different mechanisms, each producing different crystal morphologies. The acicular crystals of mullite formed from kaolinite are a consequence of the residual structure of kaolinite and formation of

mullite by exsolution of  $\text{SiO}_2$ . Crystals of mullite arising from  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixtures are initially pseudomorphic after the  $\text{Al}_2\text{O}_3$  grains until the system reaches chemical equilibrium. With subsequent growth by a solution-precipitation process (Ostwald ripening), they become rectangular with rounded ends, and elongate after longer firing times.

All the additives, except possibly the alkalies, increase the growth rate of mullite formed from  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  mixtures as the viscosity of the liquid is decreased and the diffusivities are increased.  $\text{CaO}$  and  $\text{Fe}_2\text{O}_3$  have the most pronounced effect on crystal size. The effect is not as great on kaolinite, possibly because the liquid formed is less pure owing to the impurities present in the kaolin, which can modify the viscosity effects.

Iron oxide in larger amounts changes the mullite crystal morphology in the  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  samples, making it similar to that of the kaolinite samples. Iron oxide is the only additive which will enter into solid solution with mullite in significant amounts. It is postulated that the growth in the 'c' direction is caused by a relative lowering of the surface energy of the parallel faces resulting in strong anisotropy of energies. This strong anisotropy, exhibited more extensively by kaolinite, can be due to starting structural differences and incorporation of existing impurities during the reaction.

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Table 1. Chemical Analysis of Materials

Constituent	Wt%		
	Calcined Georgia Kaolinite*	A-14	Silica Flour
$Al_2O_3$	50.60	99.6	0.06
$SiO_2$	47.22	0.12	99.80
$Fe_2O_3$	0.38	0.03	0.02
$Na_2O$	0.19	0.04	-
$K_2O$	0.19	-	-
$TiO_2$	1.40	-	0.013
$CaO$	<0.01	-	0.01
$MgO$	0.02	-	0.01
$MnO$	<0.01	-	-
$P_2O_5$	<0.002	-	-
LOI	-	0.2	0.09

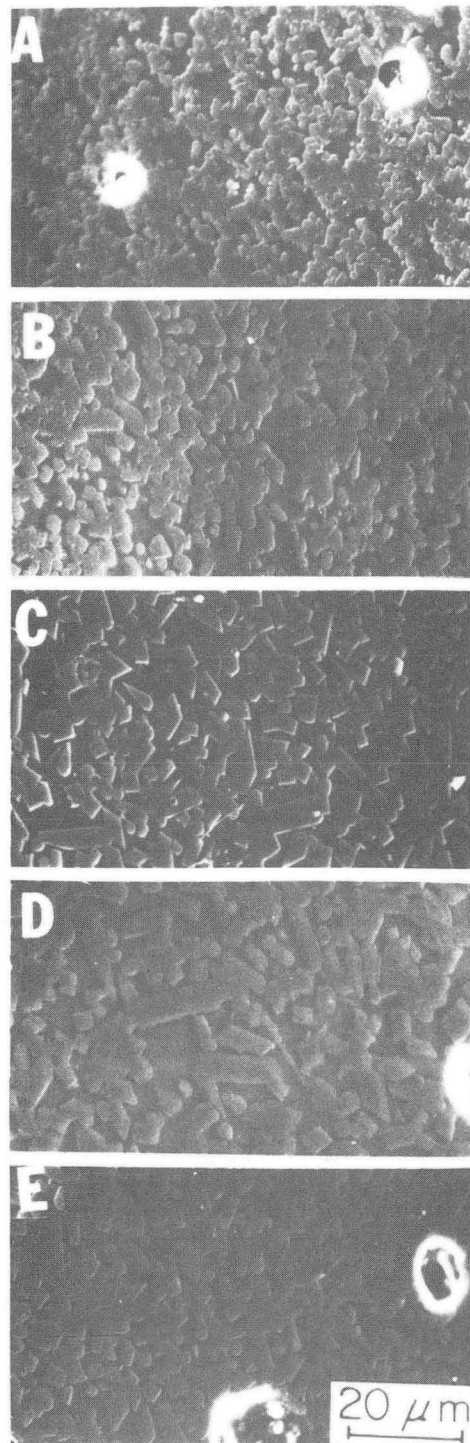
\*Chemical analysis performed by Anamet Laboratories, Inc.

Table 2. Composition of Mixtures.

%								
Mix #	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	CaO	Calcined Kaolinite
I	57.73	48.27	-	-	-	-	-	-
II	51.00	47.59	1.41	-	-	-	-	-
III	51.53	48.09	-	0.39	-	-	-	-
IV	51.53	48.09	-	-	0.19	0.19	-	-
V	50.60	47.22	1.40	0.38	0.19	0.19	-	-
VI	-	-	-	-	-	-	-	100
VII	50.70	47.30	-	-	-	-	2	-
VIII	48.78	45.57	-	5.68	-	-	-	-
IX	-	-	-	-	-	-	2	98
X	-	-	-	5.68	-	-	-	94.32

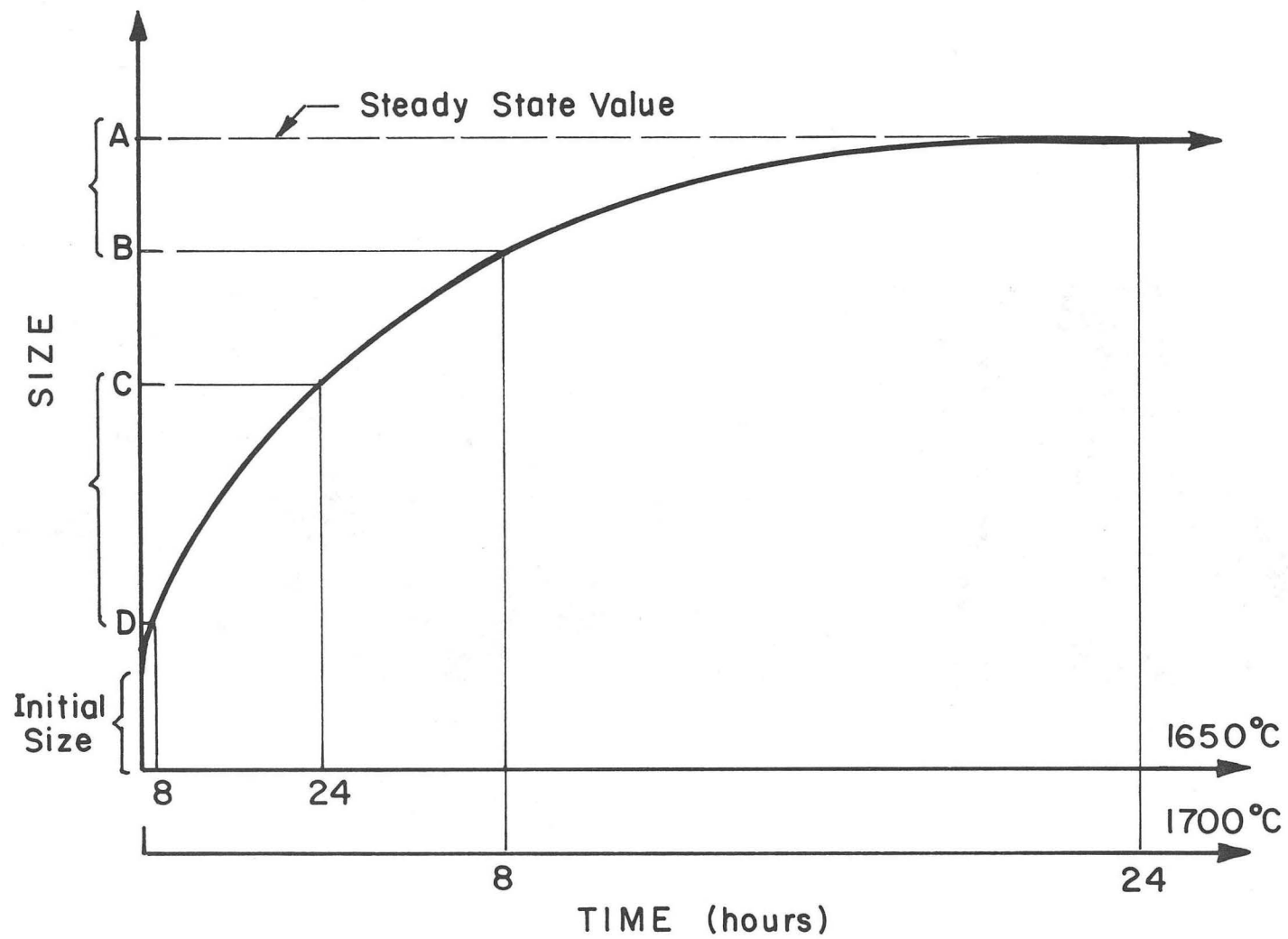
FIGURES

1.  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  compacts heat treated at (A) 1650°C/8 h, (B) 1650°C/24 h, (C) 1700°C/8 h, (D) 1700°C/24 h, and (E) 1700°C/8 h plus 1650°C/16 h.
2. Hypothetical curve for crystal size vs time at several temperatures.
3. Kaolinite compacts heat treated at (A) 1650°C/24 h, (B) 1700°C/24 h, (D) 1700°C/8 h plus 1650°C/16 h, and (E)  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  compact with additions of 1.4 wt%  $\text{TiO}_2$ , 0.38 wt%  $\text{Fe}_2\text{O}_3$ , 0.19 wt%  $\text{Na}_2\text{O}$  and 0.19 wt%  $\text{K}_2\text{O}$  at 1700°C/8 h plus 1650°C/16 h.
4.  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  compacts heat treated at 1700°C/8 h plus 1650°C/16 h with additions of (A) 1.4 wt%  $\text{TiO}_2$ , (B) 0.34 wt%  $\text{Fe}_2\text{O}_3$ , (C) 0.19 wt%  $\text{Na}_2\text{O}$  + 0.19 wt%  $\text{K}_2\text{O}$ , (D) 2.0 wt%  $\text{CaO}$ , and (E) kaolinite with 2.0 wt%  $\text{CaO}$ .
5.  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  compacts with addition of 5.7 wt%  $\text{Fe}_2\text{O}_3$  heat treated at (A) 1700°C/8 h plus 1650°C/16 h, (C) 1650°C/24 h, (E) 1700°C/24 h; and kaolinite compacts with 5.7 wt%  $\text{Fe}_2\text{O}_3$  heat treated respectively (B), (D) and (F).
6. Higher magnification of Fig. 5F.



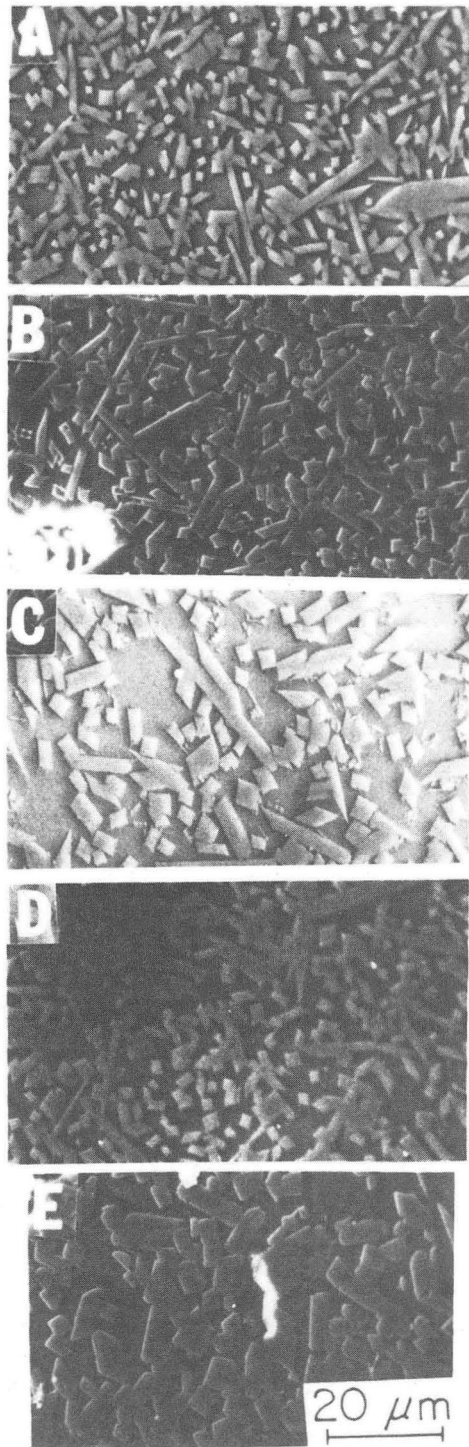
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Fig. 1



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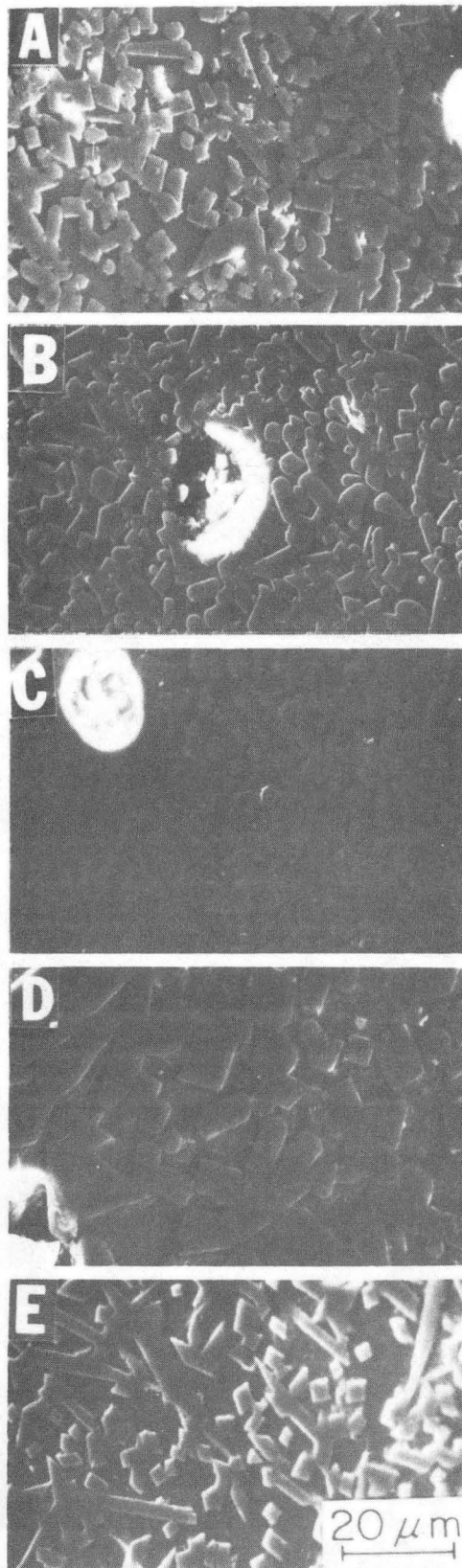
Fig. 2



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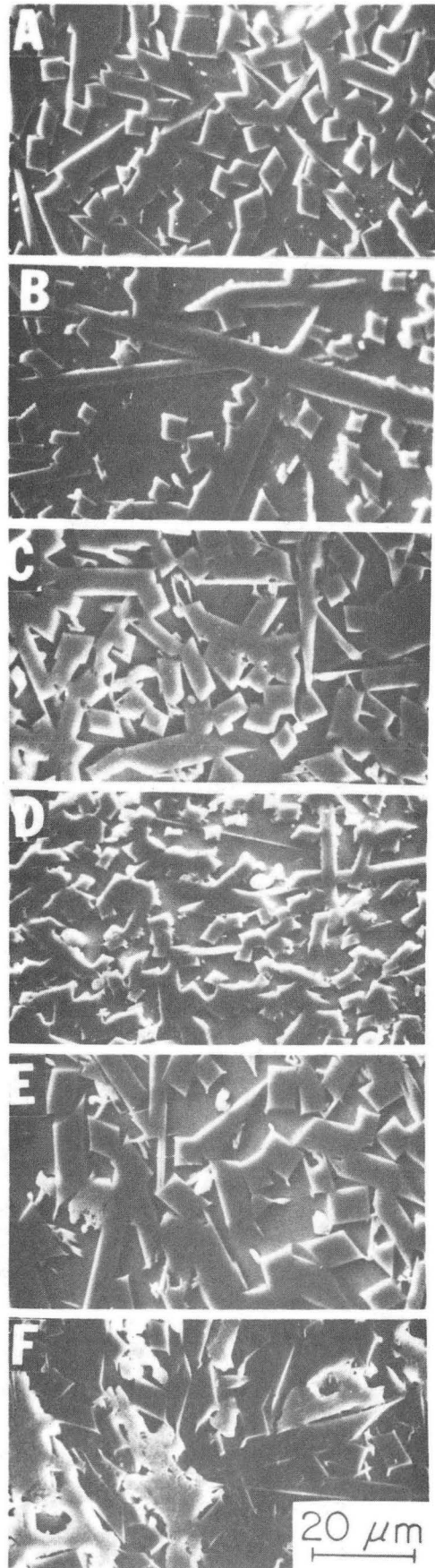
Fig. 3





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Fig. 4



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Fig. 5

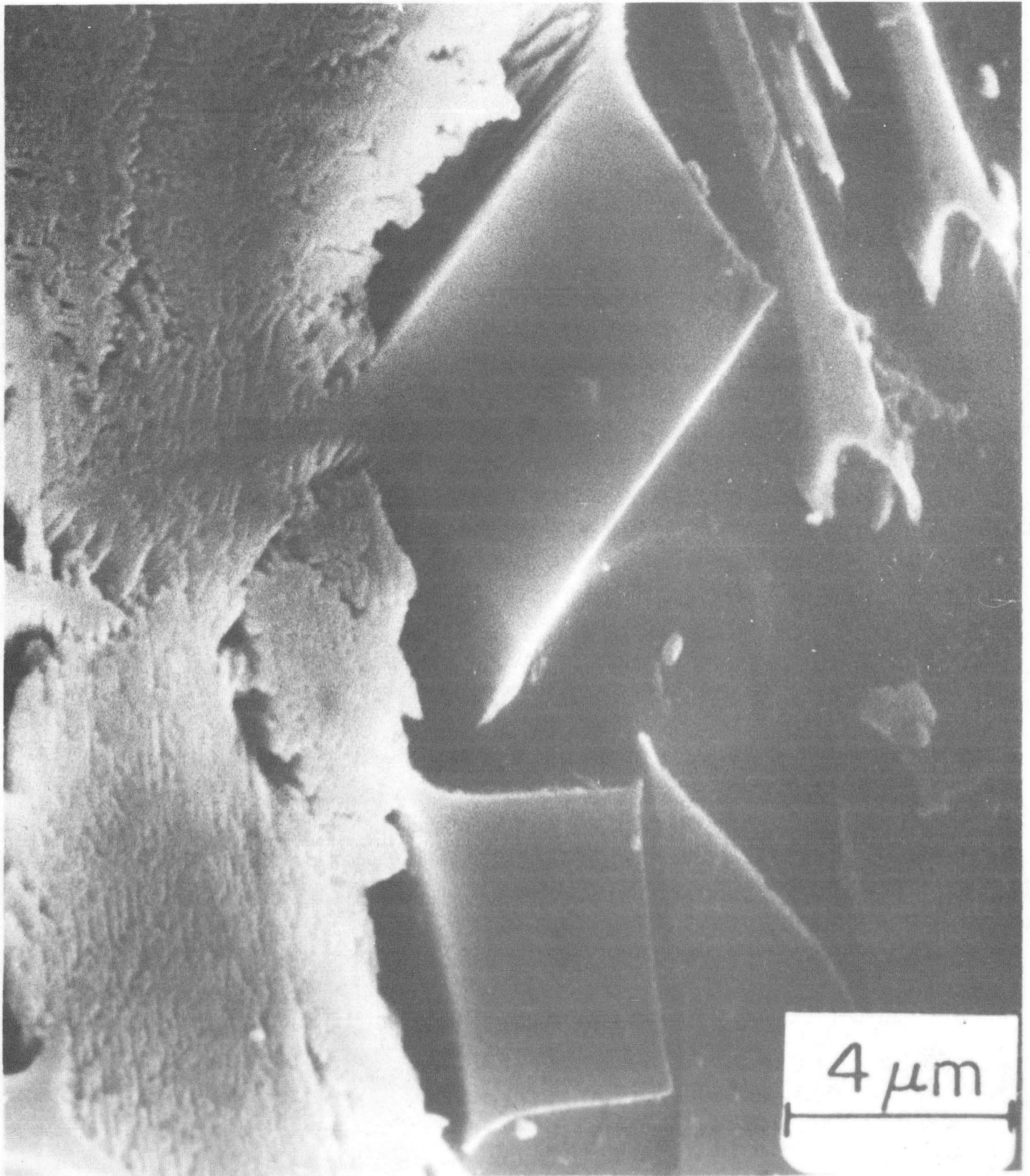


Fig. 6

XBB793-3127

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