

Chapter 6: Preparation of Compacts

6.1 Raw materials

The following powders were used.

- Sumitomo AKP-53 (α -alumina) as primary alumina constituent or as seeding in predominantly α -alumina suspensions.
- Sumitomo AKP-50 (α -alumina) as primary alumina constituent.
- Degussa alumina (γ -alumina) as primary alumina constituent.
- Grade 2 natural diamond (acid washed).
- Degussa titania as grain growth inhibitor in the alumina matrix.

Powders are characterised in detail by Msibi (1997).

6.2 Suspension of powder

Firing experiments were conducted in parallel with the development of suspension preparation routes, with the result that different routes were used during the project. Suspension preparation generally followed the sequence:

- 1) pH adjustment of suspending solution water.
- 2) Addition of dispersant.
- 3) Addition of powder.
- 4) Sonification.

Some of the steps were not used in every route; details of individual preparation routes are given in table 6-1.

Not detailed in table 6-1 is route CP. Route CP only required the mixing of suspensions prepared by routes P α or P γ and C to the required ratio of diamond to alumina, followed by additional sonification.

Preparation by route P α entailed adding powder (which tends to bring the pH down) to the suspension while maintaining pH 11. It was not very reproducible due the difficulty of measuring pH in a viscous suspension over prolonged periods of time.

Table 6-1: Preparation of suspensions.

	Route pH _α	Route HP	Route pH _α †	Route P _α	Route P _γ	Route C	
Total solids loading (% v/v)	<25 [#]	30	<30 [‡]	30	10	30	
Dispersal method	pH 4 (Nitric acid)	Hydropalat 3204	pH 11 (Ammonia)	Propionic acid	Propionic acid	1 % w/w CTAB [§]	
Dispersant/Water (% w/w)	Not applicable	1.3	Not applicable	0.85	0.85	0.75*	
Solid constituents	AKP-50	✓	×	×	×	×	
	AKP-53	×	✓	✓	✓	×	
	Degussa alumina	×	×	×	✓	×	
	Diamond	×	✓	✓	×	✓	
	Titania	In some cases	✓	✓	✓	✓	
Mixing procedure [†]	pH pre-adjustment	pH 4 with nitric acid	×	pH 11 with ammonia solution	pH 4 with nitric acid	pH 4 with nitric acid	
	Addition of dispersant	Not applicable.	✓	Not applicable.	✓	✓	
	Addition of titania	In some cases	✓	✓	✓	×	
	Addition of α-alumina seeds	×	×	×	×	✓	
	Addition of primary constituents	Alternate addition of small amounts of alumina and ammonia solution while keeping the pH close to 4 by the addition of nitric acid	Dry mixture of alumina and diamond	Alternate addition of small amounts of alumina and diamond and ammonia solution while keeping the pH close to 11 by the addition of ammonia solution	α-alumina	γ-alumina	Diamond
	Sonification	✓	✓	✓	✓	✓	✓
	pH readjustment	pH readjusted to 4 with nitric acid	×	pH readjusted to 11 with ammonia solution	×	×	×

[#] The total amount of solids used was calculated as $\frac{1}{3}$ of the initial liquid volume. After addition of nitric acid solution solids loading was therefore less than 25 % v/v.

[‡] The total amount of solids used was calculated as $\frac{3}{7}$ of the initial liquid volume. After addition of ammonia solution solids loading was therefore less than 30 % v/v.

[§] Cetyltrimethylammonium bromide.

* I.e. 0.75 % of a 1 % w/w solution of CTAB.

[†] The mixing procedure followed the order (from top to bottom) indicated.

6.3 Consolidation of suspensions

Suspensions were filter pressed by either the pressurised air or the direct force device depicted in fig 10-1 and 10-2.

In the pressurised air device pressure is supplied by compressed gas. Maximum working pressure for this press is 5 MPa, which was also the pressure used for the preparation of compacts. Two sizes of inserts were used: 35 and 8 mm diameter, which allowed the formation of compacts of the corresponding size. Filtration time for compacts that are evaluated in this work was 2 hours.

In the direct force device the load is supplied mechanical with a small press. The pressure (as experienced by the suspension) used for preparation was 20 MPa. Filtration time varied, but a minimum of 20 minutes was used successfully for AKP-53 containing suspensions, and 60 minutes for Degussa alumina containing suspensions.

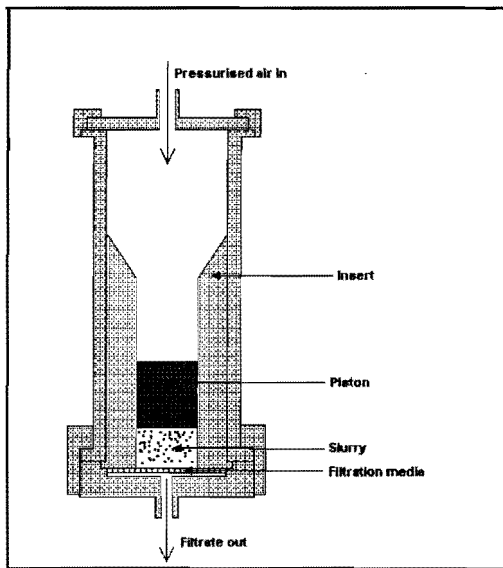


Fig. 10-1: The pressurised air device.

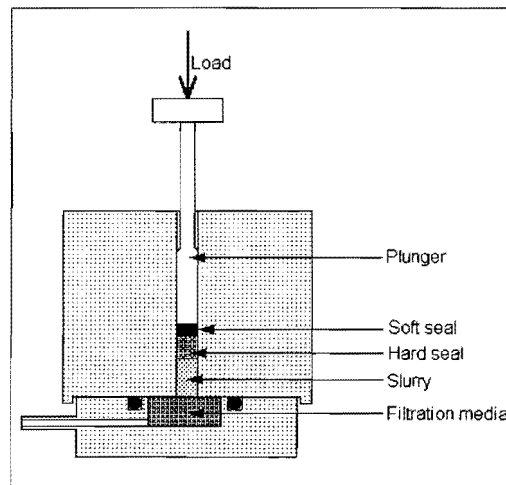


Fig. 10-2: The direct force device.

6.4 Firing

6.4.1 Introduction

An attempt was made to duplicate the results of Yeh and Sacks (1988a) (section 3.2, page 19), as they achieved high densities by sintering at low temperature in air. These attempts were unsuccessful and afterwards densification was only done by HIPping.

6.4.2 Sintering

Suspensions were prepared for sintering by route pH α . Titania (1 % m/m) was added to some samples. For sintering runs only green body formation by the pressurised air device were used. The resulting compacts were sintered in air by heating at 250 °C/h to the dwell temperature. Dwell time was 2 hours followed by natural cooling in the furnace.

6.4.3 HIPping

Encapsulation (in titanium) for HIPping and HIPping itself was conducted by Mattek[‡]. As such, precise control of the conditions was limited to the specifications given to Mattek.

Specifications were as follows:

- The pre-pressure was to be as great as possible, which amounted to 60 bar.
- The dwell temperature and pressure were to be reached as rapidly a possible.
- The dwell pressure was to be as high as possible, which amounted to 150 ± 2 MPa.
- The dwell time was to be 30 minutes.
- The compacts were to be left to cool down naturally in the HIP vessel.

A variety of mechanical methods were used to remove the titanium HIPping capsules, but immersion in warm concentrated hydrofluoric acid proved the most efficient method of removal.

[‡] Mattek, CSIR, Pretoria, South Africa.