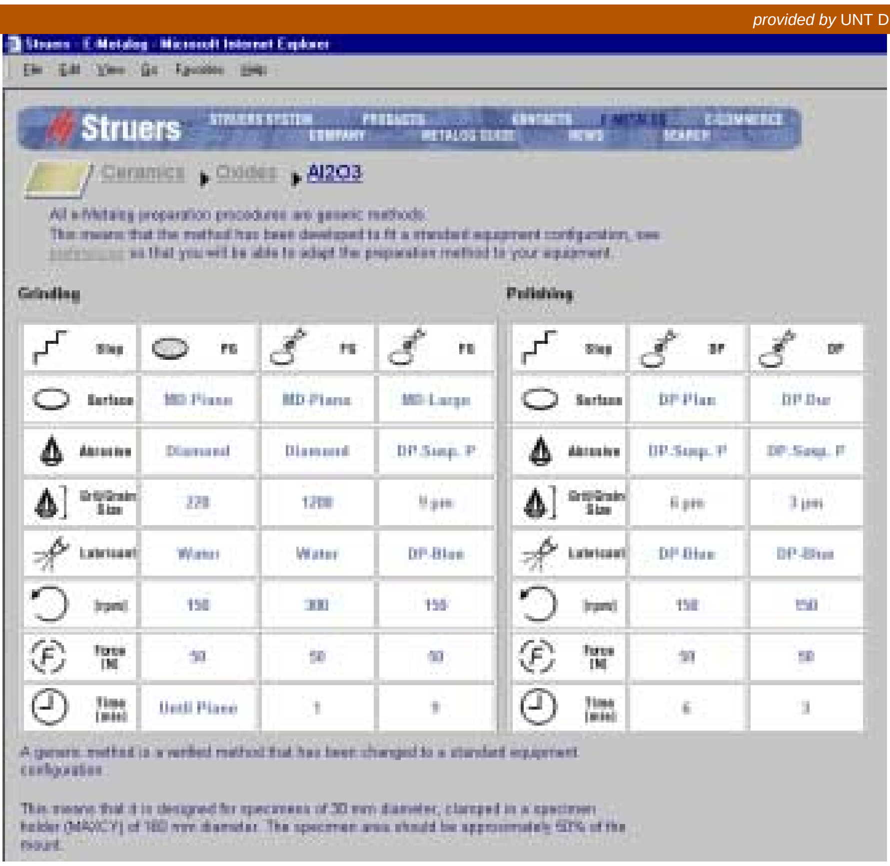
played, ready for use.

The preparation methods in e-Metalog are taken from Struers methods database IMAD, and they have been developed by specialists in the Struers application laboratories in the USA, UK, D, F, J, and in the main laboratory in Copenhagen.

Those original methods have been developed for a specific material e.g. a ceramic like Al_2O_3, the material has a defined condition and the method takes the special requirements, such as perfect edge retention, into account. The work has been done on different equipment but has been converted for e-Metalog to the same generic equipment configuration as the Metalog Guide methods, a RotoSystem with 300 mm dia disc, 160 mm dia. specimen holder, 6 x 30 mm dia. mounted specimens.

After selecting the main group ceramics and the sub-group oxides the method for Al_2O_3 is displayed



All e-Metalog preparation procedures are generic methods.
This means that the method has been developed to fit a standard equipment configuration, see [illegible] so that you will be able to adapt the preparation method to your equipment.

Grinding

Step	PG	FG	FG
Surface	MD-Piano	MD-Plano	MD-Largo
Abrasive	Diamond	Diamond	DP-Susp. P
Grit/Grain Size	220	1200	9 µm
Lubricant	Water	Water	DP-Blue
[rpm]	150	300	150
Force [N]	50	50	50
Time [min]	Until Plane	1	9

Polishing

Step	DP	DP
Surface	DP-Plan	DP-Dur
Abrasive	DP-Susp. P	DP-Susp. P
Grit/Grain Size	6 µm	3 µm
Lubricant	DP-Blue	DP-Blue
[rpm]	150	150
Force [N]	50	50
Time [min]	6	3

A generic method is a verified method that has been changed to a standard equipment configuration.

This means that it is designed for specimens of 30 mm diameter, clamped in a specimen holder (MAXCY) of 160 mm diameter. The specimen area should be approximately 50% of the mount.

e-Metalog is to be found on Struers homepage www.Struers.com.

To enter you must have a user name and a password. Fill out a short form to register and the user name and password will be send to you by mail.

A Ceramographic **Evaluation of** Chromia **Refractories** Corroded by **Slag**

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Abstract

This paper describes the ceramographic preparation of Cr_2O_3–Al_2O_3 refractory bricks and subsequent microstructural analysis to determine the corrosive effects of molten slag. The porous and friable nature of the brick, especially after exposure to the slag or its individual components, presented some problems in the preparation.

Introduction

Chromia-alumina (Cr_2O_3–Al_2O_3) refractory bricks lining the combustion chambers of gasifiers are exposed to molten slag at temperatures as high as 1600°C and pressures as high as 2.8 MPa (27 atm) gauge, for as much as two years between shutdowns. The slag penetrates and reacts with the refractory, ultimately causing an expensive failure resulting from a combination of mechanical stresses related to engineering design and thermal expansion mismatches as well as corrosion. In this study, refractory bricks were exposed to individual coal slag components in a laboratory, and compared to bricks that were removed from a coal gasifier after nearly two years of continuous operation, in order to clarify the mechanisms of refractory corrosion in a gasifier.[1]

Figure 1a: Refractory brick drilled for reaction with slag components

Most gasifiers convert coal, steam and oxygen into syngas—CO, H_2, CO_2 and byproducts—as fuel for combined-cycle turbine power plants. Gasification, in use worldwide, is a more efficient and lower-emission process than conventional coal-fired power generation. Gasifiers have been developed that use petroleum coke, black liquor from paper mills, biomass, and other organics as feedstocks. Some gasifiers produce other fuels such as methanol and methane, or chemicals such as ammonia.[2,3,4]
The feedstock typically contains oxide mineral components such as SiO_2, FeO and CaO, which form an amorphous slag that flows slowly down the refractory walls.

High-chromia compositions of refractory bricks consistently outlast other compositions in gasifier applications.[5,6] Cr_2O_3 is generally less soluble than Al_2O_3 or MgO in CaO–SiO_2 slags.[7] Aurex® 90 bricks,[ii] nominally 90 mass% Cr_2O_3 and 10% Al_2O_3, were used in this study. The chromia content is roughly 86% of the mole fraction in this composition, resulting in a microstructure that is predominately a solid solution of (Cr, Al)$_2$O$_3$. Chromia-alumina bricks are also available in 75, 60, 40 and other mass percentages Cr_2O_3, and most have about 15 volume% porosity. Chromia-zirconia (Zirchrom™)[iii] and magnesia-chromia (“magchrome”)[iv] bricks are also used in similar applications.

Figure 1b: Cross-section of the drilled refractory brick after thermal treatment and backfill with epoxy

Experimental Procedure

Heat Treatment

Sections of virgin refractory bricks were drilled to create an array of cavities, 15 mm diameter by 15 mm deep, as shown in Figure 1a. The cavities were filled with various combinations of SiO_2, FeO and CaO powders, and an actual slag powder from a gasifier. The slag contained 46 mass% SiO_2, 18% FeO, 19% Al_2O_3, 10% (Ca, Mg)O, and other oxides as the balance. The specimens were heated in a sealed tube furnace to 1550°C for 24 hours, in a flowing reducing gas of 50:50 Ar-CO that had been bubbled through water. The reducing atmosphere prevented the metastable FeO from oxidizing to a more stable form, i.e. Fe_2O_3.

All the powders penetrated and reacted with the refractory during the furnace cycle.[8]

The spent refractory bricks removed from the gasifier were examined as received, without any further thermal processing. The virgin refractory brick was also examined without any oxide or slag treatment, as an experimental control.

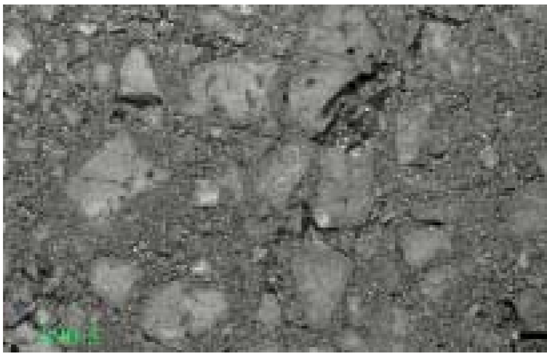

Figure 1c: Polished cross-section of a 90% Cr_2O_3 refractory brick. The primary phase is a solid solution of (Cr, Al)$_2$O$_3$

Ceramography

The virgin refractory, the reaction cavities in the refractory section - cups - and the spent bricks were ceramographically sawed, mounted and polished by methods similar to those described by Asakura and Takahara,[9] Podwórny,[10] Chinn[11] and many others. The specific details are given below.

Cutting and Mounting

The cups were first filled with an epoxy resin to stabilize the slag-refractory interface regions and prevent loss during cutting, and then cross-sectioned with a water-cooled diamond wafering blade. Oil coolant is difficult to remove completely from materials that have a continuous network of pores, and therefore was not used. The spent refractory samples were encapsulated with epoxy resin to reinforce and stabilize the fragile slag-refractory zone before sectioning, as shown in Figure 1b.

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[i] Reference herein to any specific commercial product, process or service by trade name, trademark, manufacturer or otherwise, does not necessarily constitute or imply its endorsement, recommendation or favoring by the United States Government or any agency thereof or its contractors or subcontractors

[ii] RHI Refractories America, Inc., Pittsburgh, Pennsylvania, USA 15219

[iii] Compagnie de Saint-Gobain, Paris, France

[iv] Exolon-ESK Company, Tonawanda, New York, USA 14150

Table I: Automatic preparation steps for chromia-alumina refractories on the Abrapol automatic grinder/polisher

	Grinding				**Polishing**	
Step	**1**	**2**	**3**	**4**	**1**	**2**
Surface	MD-Piano 220	MD-Allegro	MD-Allegro	DP-Plan	DP-Dur	DP-Dur
Abrasive	Diamond	DP-Suspension	DP-Suspension	DP-Suspension	DP-Suspension	DP-Suspension
Grain size		15 µm	9 µm	6 µm	3 µm	1 µm
Lubricant	Water	DP-Blue	DP-Blue	DP-Blue	DP-Blue	DP-Blue
Speed [rpm]	150	150	150	150	150	150
Force [N]	100	100	200	200	200	100
Time [min]	Until plane	5	5	10	5	5

The cup cross-sections were cut to approximately 20 x 20 x 45 mm, dried in an oven, and degassed at 74 kPa negative pressure. Sections of 60 mm (outside diameter) polyvinyl chloride (PVC) plastic pipe were used for ring molds. The large mold size was selected to contain the entire reaction region of each cup, and to match the dimensions of the specimen holder. The inside walls of the molds were knurled and coated with PVC primer (purple dye, methyl ethyl ketone and other organic solvents) to strengthen the bond with the epoxy resin. Masking tape was used to seal the bottom of the mold and form a cup. The spent refractory specimens were cold-mounted in epoxy resin and vacuum impregnated at 74 kPa negative pressure for ten minutes at ambient temperature. The virgin refractory sections were compression-mounted in a metallographic mounting press with glass-filled diallylphthalate, as shown in Figure 1c.

Grinding and Polishing

Planar grinding was carried out on a permanently bonded, 220-grit MD-Piano[v] diamond disc on an automatic machine. Fine grinding was performed with hard composite MD-Allegro[v] discs with 15 and 9 µm diamond suspensions. A hard napless cloth with 6, 3 and 1 µm polycrystalline diamond suspension was used for polishing. All the diamond suspensions were water-based. The specimens were carefully cleaned between each step of grinding and polishing to prevent coarser particles from being carried over to the next step. Oxide polishing compounds were not used for final polishing due to the adverse effect of the residue on the x-ray microanalysis. Because the hardness and matrix of each sample differed significantly and the mounted surfaces were unusually large, the grinding and polishing times ranged from 1 to 20 minutes and the applied force varied from 100 to 200 N.

The specimens were cleaned in a solution of Micro-90®[vi] in water in an ultrasonic bath. Cleaning times were limited to about 1 minute to prevent pullout of the grains. The specimens were removed from the ultrasonic cleaner, rinsed with distilled water, rinsed with ethanol, and dried under a heat gun.

Microscopy

All specimens were examined by light microscope and scanning electron microscope (SEM).

The energy-dispersive x-ray spectrometer of the SEM was used to generate an element map around each cup to determine the depth of penetration by the slag or its components.

Results and Discussion

Preparation Difficulties

Preparation of refractory ceramics presents several difficulties not normally encountered with densified ceramics and metals, due mostly to the high porosity and weak bonds between grains. Penetration of the refractory by the oxides and slag can further weaken the bonds. The result was that the refractories had numerous pullouts, and a very dull surface after the final polishing step. Vacuum impregnation with castable epoxy resin minimized the pullouts, by outgassing the pores and filling the voids with resin, but had limited effectiveness. The PVC ring minimized the edge-rounding effect.

Slag Penetration and Reaction Oxides

In Figure 2, the silica powder did not react with the chromia refractory to form a new phase, as expected from the SiO_2–Cr_2O_3 phase diagram. From the x-ray map, the silicon did penetrate some 5 mm into the refractory matrix. An x-ray diffraction analysis showed that only the original $(Cr, Al)_2O_3$ sesquioxide phase was present in the refractory.

The silica-refractory interface remained intact.

The calcia reacted with the refractory to form a low-melting $Ca([Cr, Al]O_2)_2$ phase at the ox-

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[v] *Struers A/S, DK-2610 Rødovre, Denmark*

[vi] *International Products Corporation, Burlington, New Jersey, USA 08016*

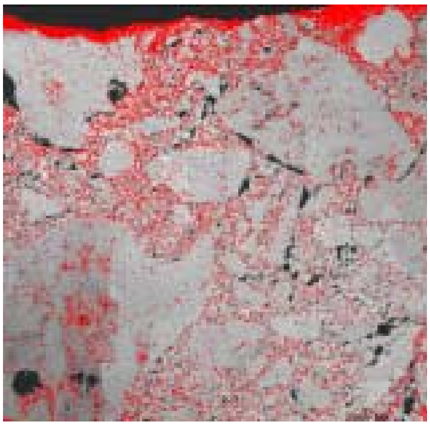

Figure 2: Silica (Si penetration in red) attack on the 90% Cr_2O_3 refractory cup

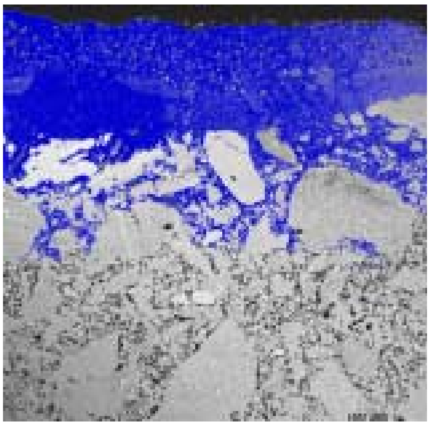

Figure 3: CaO (Ca penetration in blue) attack on the refractory cup

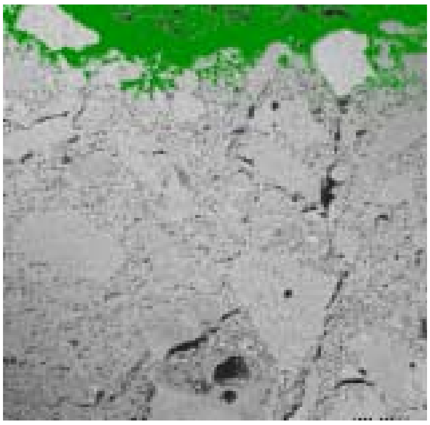

Figure 4:
FeO (Fe penetration in green) attack on the refractory cup

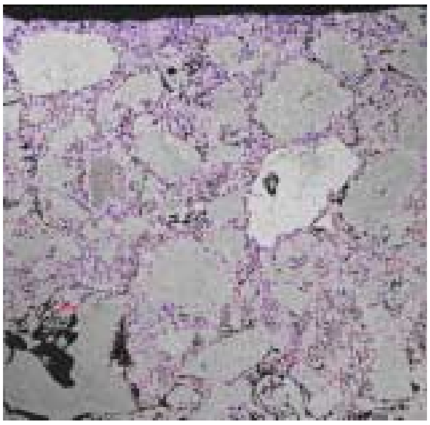

Figure 5:
Silica (penetration in red) and CaO (blue) attack on the refractory cup

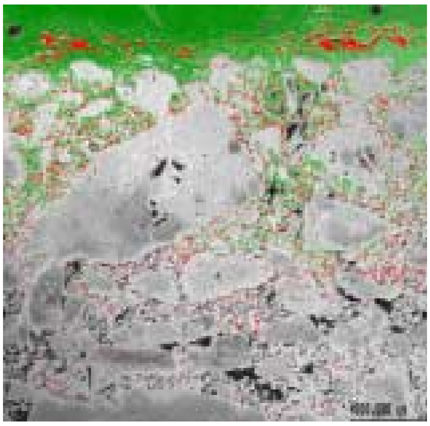

Figure 6: Silica and FeO (Fe penetration in green, Si in red) attack on the refractory cup

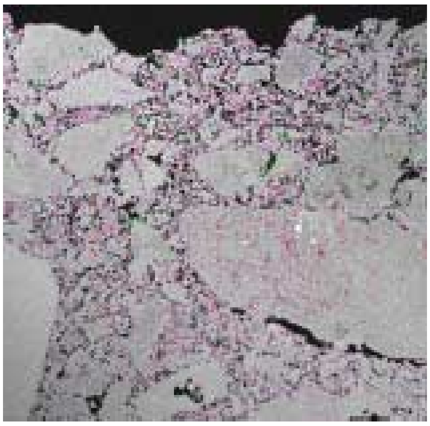

Figure 7: Silica, CaO and FeO (Fe penetration in green, Si in red, and Ca in blue) attack on the refractory cup

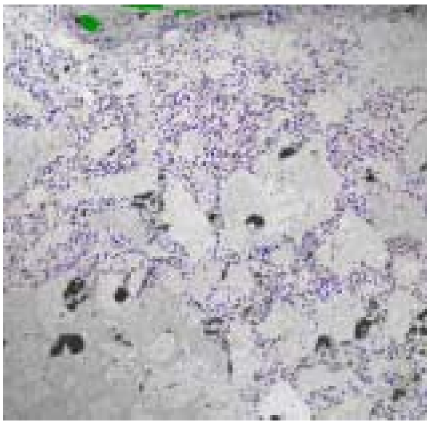

Figure 8:
Actual slag (Si penetration in red, Ca in blue, Fe in green) attack on the refractory cup

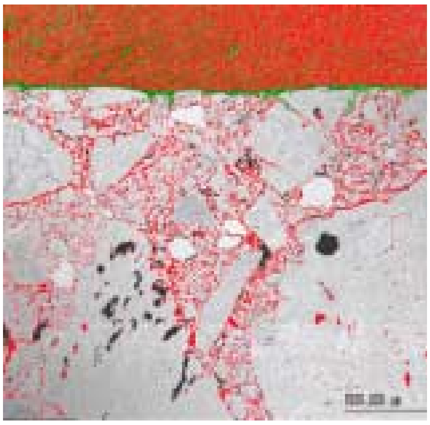

Figure 9:
Actual slag (Si penetration in red, Fe in green) attack on the spent refractory from the gasifier

ide-refractory interface, in Figure 3. The reaction was evident from XRD results and the discoloration and texture differences in the microstructure around the cup. The calcium also penetrated the refractory, but not as much as the silica did. The $Ca([Cr, Al]O_2)_2$ phase beneath the porous top layer appears dense and potentially could prevent other species from penetrating into or reacting with the refractory.

The FeO (wüstite) reacted with the refractory to form a spinel phase, $Fe(Cr, Al)_2O_4$, which is apparent in the top layer of the microstructure in Figure 4. The iron did not penetrate the refractory significantly, but the oxide-refractory interface changed dramatically. The original drilled cup profile surfaces were obliterated by the spinel reaction.

In the dual oxide test where equal masses of silica and calcia powders were heated in the cup, the silicon and calcium together penetrated deep into the refractory in Figure 5, but did not react with the refractory to form a new phase. The wall of the cup was barely affected.

The silicon and iron had deep penetration in the silica-FeO test shown in Figure 6, similar to the silica-calcia dual oxide test.

The FeO reacted with the refractory to form the $Fe(Cr, Al)_2O_4$ spinel phase, and the original cup surface changed significantly in both color and texture. The silica and FeO initially reacted at about 1250°C to form a low-melting phase.

In the multiple oxide test where equal masses of silica, FeO and calcia powders were used, the Si and Ca again penetrated deep 4 into the refractory while most of the FeO reacted to form the Fe(Cr, Al)$_2$O$_4$ spinel phase at the interface. The color, texture and dimensions of the interface changed, although not as much as in the two previous tests that employed larger amounts of FeO. The microstructure is shown in Figure 7.

Slag

The actual slag penetrated and reacted with the cup in Figure 8. The result was very similar to the multiple oxide test in terms of penetration. The Si and Ca penetrated deep into the refractory while the FeO reacted to form the Fe(Cr, Al)$_2$O$_4$ spinel phase at the interface. The interface region melted during the reaction, and solidified into a porous top layer and a dense layer underneath. The spent refractory was highly eroded on the surface and also had a 1–2 mm thick slag layer adhering to the surface, in Figure 9. Similar to the multiple oxide and actual slag cup tests, the Si and Ca again penetrated deeply into the refractory while the FeO reacted to form the Fe(Cr, Al)$_2$O$_4$ spinel phase near the surface.

Conclusions

Ceramographic analysis showed that the corrosive effect of slag on chromia-alumina refractory brick was not necessarily equal to the sum of the effects of the components of the slag. Silica, the primary component, penetrated the open pores easily but did not react with the original (Cr, Al)$_2$O$_3$ phase. The depth of penetration by calcium and iron, and the extent of the reaction between the calcia and the refractory, appeared to depend upon the presence of silica in the slag. Calcia and especially FeO tended to react with the refractory at the slag-refractory interface to form compounds that melted at lower temperatures than the oxide or the refractory. The new phase appeared to lessen further penetration by the slag components into the refractory.

Acknowledgements

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The **story** of the **first Abra-**machines

Johanna Löttinger,
Lambda Instrument AB

1.000.000.000 revolutions

That is how many times a rolling bearing may rotate during its lifetime. Did you also know that an average household in the western world today contains more than 150 rolling bearings? They are everywhere, in your kitchen appliances, in your washing machine, in your computer and in your car. There are even miniature rolling bearings in the darts that professional dart players use! Most of these rolling bearings probably come from SKF AB, and the quality control of the steel in the bearings has been carried out on an Abra-system from Struers for almost thirty years. What you probably did not know, is that SKF was deeply involved in the development of the Abra-machines, and that the first ever Abrapol and Abraplan was delivered here in the early 70’s!

The Struers symposium in 1970

It all started back in February 1970. Struers then invited a number of Swedish customers to a symposium, named “Metallographic sample preparation for the 70’s”. The participants came from a number of well-known Swedish industries, such as Volvo AB, SKF AB, Sandvik AB and Avesta Sheffield AB. At this date, most metallographic laboratories were doing manual preparation of the samples, which was difficult, time-consuming and boring.

The topics for the symposium were:

- What type of machines do you need in the future?
- Can we use existing equipment or do we need to develop new machines?
- How can we transform these ideas to serial made machines?

Many wild ideas were discussed, and one of the wildest was a machine for automatic sample preparation. The idea came from SKF Steel AB (now Ovako Steel AB) in Hofors, who were willing to invest time and money on development in cooperation with Struers.

The original idea was to fix the samples in one position, and then have the different surfaces for grinding and polishing on rotating arms and let them work on the samples in consecutive steps. This idea was quickly abandoned, and Struers started the design of a semi-automatic polishing machine instead. The first drawings were sent to Mr Hans Söderström in Hofors in November 1970. After this an extensive correspondence took place, and in March 1971 the first quotation for the machine was sent to SKF Steel AB. The price quoted was 12 100 SEK (10 SEK » 1 USD). The first prototype of an Abrapol was delivered in July 1971. Specimen holders were ordered from a local mechanical firm at a cost of approximately 400 SEK a piece.

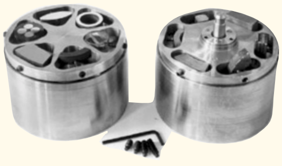
The development of the Abraplan

From the very beginning SKF Steel AB claimed that a coarse grinding machine was necessary to increase the speed of the plane grinding, but Struers insisted that plane grinding on paper would be sufficient. In September 1971 SKF Steel AB expressed their satisfaction with the Abrapol, but once again stressed the importance of a separate machine for fast plane grinding. Struers then decided to continue the design of the existing polishing machine, but to also develop a new plane grinding machine.

The first plane grinding machine, better known as Abraplan, was


One of the first prototypes in Struers lab in Copenhagen


The first Abrapol prototype delivered to SKF


The original specimen holders, especially made for SKF Steels samples