

# Surface Potential Influence on Optical Glass in the Polishing Process

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In the past years, research projects on the impact of chemical process parameters on the results of the polishing process of optical glass became more and more important. In the AiF project "Chemical Impact on Optical Glass in the Polishing Process" a wide range of chemical influences were determined and correlated with mechanical parameters analyzed by the project partner Fraunhofer Institute of Production Technology in Aachen.

The conclusions of the project<sup>1</sup> are in detail:

- A. The glass exposes a virgin surface to the polishing tool during the entire duration of the process.
- B. Analysis of the polishing fluids shows a strong relation between the solubility products (especially of the silicates and their aqueous associations in the suspensions) and the abrasion depth of the glasses.
- C. The presence of dissolved ions originating from the glass surface has a strong influence on the agglomeration behavior of the polishing agent in the solution.
- D. The presence and hardness of agglomerations cause a negative polishing quality. Coagulated particles are dispersed again by the pressure applied by the polishing tool, thereby absorbing energy and decreasing the efficiency of the process.

For a subsequent project the following hypothesis is stated:

The agglomeration of particles in the polishing solution is the predominant chemical parameter. It is determined by the complex solubility equilibria and the surface charges of the solid agents in the polishing solution.

This project performed at the Department of Mineral Engineering is concentrating on the electro-chemical aspects of the process in combination with the energy demand. Properties, i.e. the  $\zeta$ -potential of the polishing agent, the grain size distribution and its alteration during the process, and the surface influence of the polishing pads, will clarify the relation between surface properties and the mechanical polishing behavior.

## Introduction

In the production of optical applications, polishing is the process step determining the production time, production costs, and therefore the production economy. Several projects were concerned with the optimization of the time demand and process reliability of the finishing process. The focus of the past years was laid on the impact of the chemical parameters which were related to mechanical effects from previous project results.

### The chemical material removal in the polishing process

Several theories are accepted as the mechanisms of the polishing process<sup>2,3</sup>. In detail, these are: mechanical friction and wear mechanisms, plastic flow, chemical and tribo-chemical

mechanisms. However, a detailed quantification of the contributions of the individual mechanisms is not yet possible.

In earlier publications, the impact of the mechanical system on the synchro-speed process, e.g. the load, number of revolutions, was shown<sup>4</sup>. In a further publication, the complex system of the chemical parameters influencing the process quality was explained<sup>5</sup>. The interaction of the suspension and the glass was discussed in the latest paper<sup>6</sup>. In the present paper, the content of a future project is described concerning the scientific links between chemical and mechanical impacts in relation to the electrochemical aspects.

### Alteration of the polishing suspension

In order to understand the chemical interactions and their consequences on the polishing process, six different optical glasses (code names N-BaSF64, N-LaK8, N-SK16, N-FK51, SF6, KG1) were polished with a synchro-speed polishing machine with polyurethane pad and Opaline<sup>®</sup> (CeO<sub>2</sub>) as polishing agent at 1000 r.p.m. and 1 bar for 10 min at the Fraunhofer Institute of Production Technology (IPT) in Aachen<sup>4</sup>. The observed parameters are the mass loss per surface area and the surface roughness. Six lenses of each glass were polished with the same suspension during one run of polishing experiments. The mass loss, roughness of the glass surface, as well as the pH value, temperature, electrical conductivity, and chemical composition of the polishing fluid were observed.

The surface roughness ( $R_a$  and RMS) for all experiments was approx. 1 to 1.5 nm. Depending on the different compositions of the glasses, the pH value of the fluid increased to higher values (initial pH value 5.5). The electrical conductivity and the temperature did not change significantly during the process. During the alteration of the suspension, the silicon and the calcium content increased. As soon as the equilibrium of the Ca content was reached, the slope for the Si content started to increase again. Such a behavior is a clear indication of the involvement of a solubility limit in the process (i.e., a Ca-Si containing precipitate is formed). At the very point where the limit is reached, as disturbance of the process is reflected by a decreased abrasion depth.

As an example, figure 1 shows the atomic absorption spectroscopy results of the suspension centrifugate for N-BaSF64 and N-FK51 lenses which were polished for 10 min each.

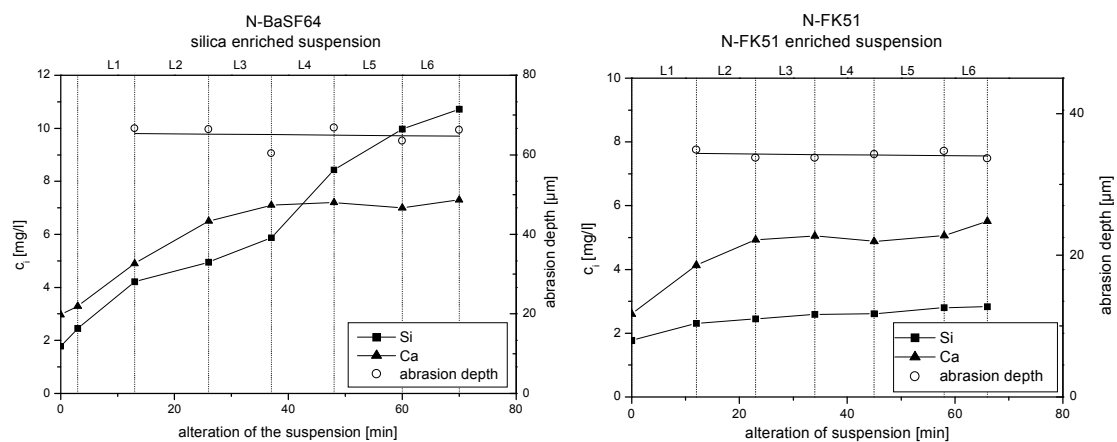


Fig. 1. Si and Ca concentrations of the polishing fluid (analyzed by atom absorption spectroscopy) and abrasion depth of N-BaSF64 and N-FK51; polishing time 10 min for each sample

#### Corrosion tests of optical glasses exposed to solutions with polishing agent

In order to clarify the chemical interactions, corrosion tests were conducted<sup>7</sup> with the same six optical glasses. The glasses were cut in small chips (2 x 2 x 0.2 cm) by a precision blade and were exposed to the corrosion medium (surface to volume-ratio = 0.06 cm<sup>-1</sup>). In order to keep the pH value constant, buffer solutions were added to the corrosion medium. For pH 7.2 the buffer contained 1.007 g 30% HCl and 3.775 g TRIS (trishydroxymethylaminomethane) in 2500 ml distilled water<sup>8</sup>. Two corrosion scenarios were analysed: i) distilled water with pH buffer solution and ii) distilled water, pH buffer, and the polishing agent (60 g/l OPALINE<sup>®</sup>). The solution was kept 70 °C and not stirred (for experimental setup see fig. 1). The pH value was controlled on a day-to-day basis, and titrated with HCl or NaOH in case of deviations. Depending on the durability of the glasses, once or twice a week the mass loss of the dried samples was measured after rinsing with distilled water and acetone.

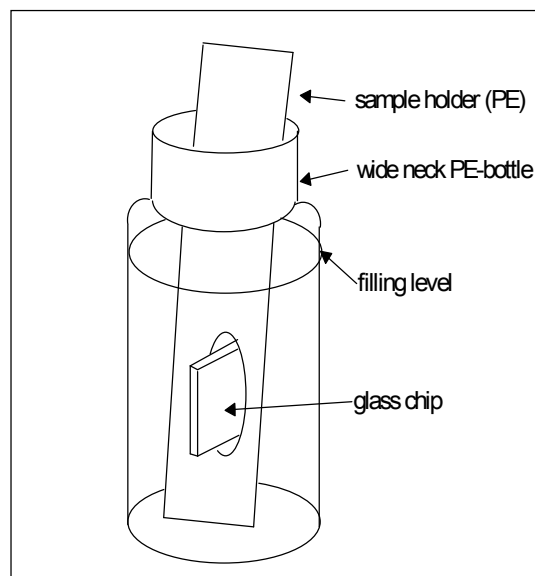


Fig. 2. Experimental setup of the corrosion tests after<sup>8</sup>

The experimental results of the corrosion tests of N-FK51 and SF6 are shown in fig. 3. The dissolution rate  $r$  (mg·cm<sup>-2</sup>·d<sup>-1</sup>) is determined by the linear regression of the mass loss  $q$  (mg·cm<sup>-2</sup>) over the corrosion time  $t$  (d). Generally the dissolution rate in water without OPALINE<sup>®</sup> is smaller than the dissolution rate in water with the polishing agent by a factor of 1.5 to 4.

## Discussion

Since the testing parameters were the same for both corrosion scenarios, the polishing agent has to be responsible for the greater dissolution rate  $r$ . Since  $\text{CeO}_2$  itself has an extremely low solubility, hence does not interfere directly with the solution chemistry, we may conclude that the parameter for the increased dissolution rate is the surface coverage of the OPALINE<sup>®</sup> grains by adsorbed ions of the corrosion products. These ions are bound to the surface of the grains thus decreasing the ion concentration of the corrosion solution. After the principle of le Châtelier the equilibrium of the dissolution reaction is shifted towards the products.

In the polishing process, the glass removal dissolves within process duration (fig. 1). Like in the corrosion tests, OPALINE<sup>®</sup> is expected to enhance the dissolution during the initial stages of the polishing process. Only if the solution reaches chemically constant conditions, it is ready for use. This agrees well with the observation in industrial practice that a polishing solution has to be “conditioned” before use by polishing dummy samples for a certain period. After an extended period of use, certain solubility limits are exceeded, and the solution will turn ineffective. These mechanisms critically depend on glass composition, pH value, and water hardness.

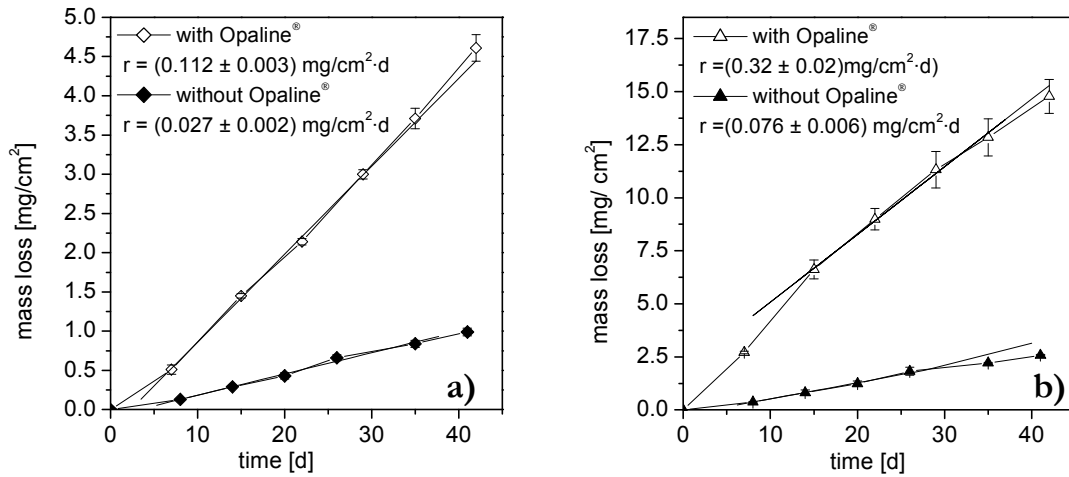


Fig. 3. Dissolution behavior of optical glasses in terms of mass loss versus test duration; a) N-FK 51 and b) SF 6

## Objectives of a new research project

As a consequence of the reported results, the objective of a new project is the investigation of the surface potential ( $\zeta$  potential) of the polishing agent and the glass surface. The measurements comprise the quantification of the adsorbed ions on the glass and  $\text{CeO}_2$  surface and detailed determination of the mechanisms of adsorption. A first task is the observation of the present process parameters. In a second step, these parameters, e.g. pH value, ion concentrations from the glass removal, and dispersion agents, will be optimized.

It has been shown<sup>1</sup>, that the electrochemical surface potentials are directly related to the electrical power demand during the polishing process<sup>4</sup>. Agglomerated and suspended polishing solutions will be compared in order to determine the loss of machine energy by the “crushing” of agglomerates.

In this way the upcoming project will provide a scientific link between the mechanics and the chemistry of the polishing process.

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<sup>2</sup> Cook, L.M., J. Non-Cryst. Solids **120**, p. 152-171 (1990).

<sup>3</sup> Kaller, A., Glastechn. Ber. **64**, p. 241-252 (1991).

<sup>4</sup> Hambücker, S., AiF, No 10607 N (1998).

<sup>5</sup> Dahlmann, U.; Groß, S.; Hambücker, S.; Conradt, R. in *74. Conf. of the Germ. Soc. of Glass Technology, Ulm*, 2000, p. 62-65.

<sup>6</sup> Dahlmann, U.; Groß, S.; Conradt, R. in *XIX Intern. Congr. on Glass, Edinburgh*, 2001, p. F1 76-77.

<sup>7</sup> Wyart, C., Institute of Mineral Engineering, Dissolution behavior of optical glasses in aqueous solutions (2001).

<sup>8</sup> Groß, S.; Dahlmann, U.; Conradt, R. in *5<sup>th</sup> ESG Conference, Prague*, 1999, B1 p. 65-72.