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USE OF EGD FOR THE CHARACTERIZATION OF FINING PROCESSES OF GLASS MELT

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The control and optimization of the glass-making process is a serious problem in glass production. An EGD procedure involving high-temperature vacuum extraction and total pressure measurement allows determination of the fining state (Läuterzustand) and fining behaviour (Läuterverhalten). The fining state is characterized by the contents of CO_2, N_2 and other gases from the batch reactions, and gas inclusions. Oxygen evolved in a second step is characteristic for the fining behaviour.

Bubbles in the final product are a serious problem in glass production, and particularly in the production of optical glass and television tubes. The control and optimization of the gas content in glass-making processes are much more important today than ever before.

Analytical determination of the gas content and the degassing behaviour requires great technological and financial effort. The mass-spectrometric or gas chromatographic methods used at present are relatively time-consuming, due to the complicated sample preparation and the long starting period [1].

By comparison with a detailed mass-spectrometric investigation of the high-vacuum degassing of a glass melt by heating, we tested the possibility of determining the fining stage of a glass sample by means of total pressure mesurements (evolved gas detection, EGD).

Experimental

The simple EGD apparatus shown schematically in Fig. 1 was successfully employed for characterization of the fining stage of container glass. With such equipment it is possible to investigate special types of glasses merely via the total-pressure change with a simple high-vacuum gauge.

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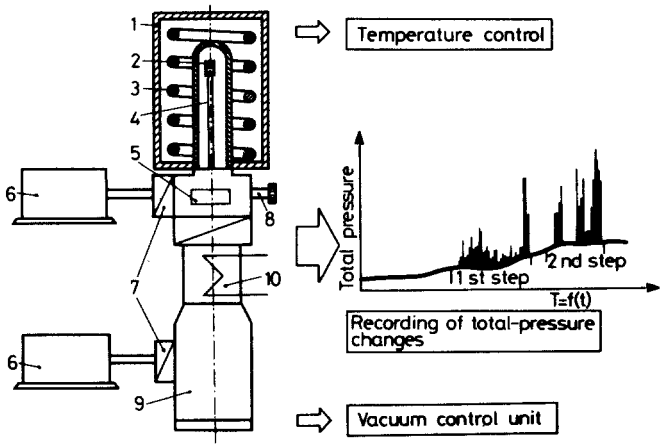

Fig. 1 Schematic diagram of the EGD apparatus.

1 Furnace,	6 Rotary pump,
2 Sample,	7 Valve,
3 Tube,	8 Needle valve,
4 Thermocouple,	9 Oil diff. pump,
5 Gauge,	10 Multicoolant baffle

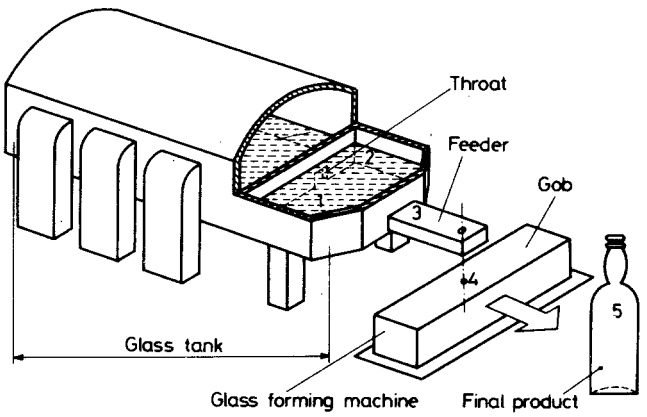

Fig. 2 Sampling positions in a glass tank

Results

The insert of Fig. 1 shows an EGD diagram. Each spike on the curve indicates the burst of a single bubble; the height of a spike corresponds to the amount of gas. A mass-spectrometric study demonstrated that the first step in the spontaneous

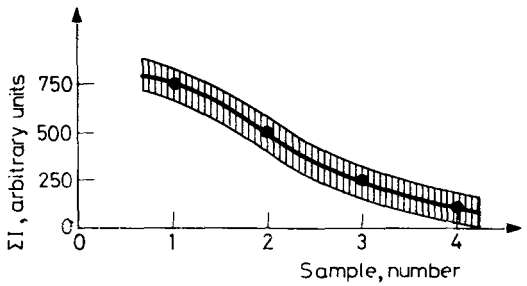

Fig. 3 Change of the summary gas content (ΣI) as a function of sampling position

degassing (bubbling) of a container glass is characterized by the evolution of CO_2, together with N_2, H_2O, etc., and the second step by the evolution of O_2 bubbles. The temperature ranges and the gas compositions of these steps are specific for a particular glass type. A rough measure for comparison purposes is the sum of peak heights $\sum I$ in a certain temperature interval, e.g. the first or second step in Fig. 1 (insert).

With our EGD technique we found that an important factor for the gas content of the sample, and consequently for the discussion of the results, is the position of sampling in the process of melting and forming of glasses. Figure 2 illustrates from which positions in a glass tank the samples were taken. From Fig. 3, the differences in gas content at different positions are obvious.

Furthermore, we detected a strong variance in the gas content at different times of sampling. Thus, the interpretation will be even more complicated. Nevertheless, the first results promise useful means of rapid characterization of (i) the degree of fining, i.e. the content of residual gases originating from the raw materials and air inclusions; and (ii) the effect of bubble-forming additives on the progress of fining.

References

1 E. Hartung, K. Heide, H.-G. Schmidt, Silikattechnik, 38 (1987) 309.

Zusammenfassung — Die Kontrolle und Optimierung des Gasgehaltes von Glasschmelzen ist eine wichtige Voraussetzung für eine gleichbleibende Qualität der Glasprouktion. Eine EGD-Technik mit Hilfe der Hochvakuumheißextraktion und Gasdruckmessung erlaubt, den Läuterzustand und das Läuterverhalten einer Glasprobe zu bestimmen. Der Läuterzustand wird durch die Gase aus den Gemengereaktionen und Gemengeeinschlüssen (CO_2, N_2 u. a.) bestimmt. Das Läuterverhalten ergibt sich aus der Menge des in einer zweiten Stufe extrahierbaren Sauerstoffs.

Резюме — Контроль и оптимизация процесса получение стекла представляют собой серьезные проблемы. Метод обнаружения выделяющегося газа, проведенный с помощью высокотемпературной вакуумной экстракции, и измерения общего давления позволили определить состояние и режим очистки. Состояние чистоты охарактеризовано содержанием двуокиси углерода, азота и других газов, образующихся при реакции шихты, а также газовых включений. Кислород, выделяющийся на второй стадии, является характерным для режима очистки.