Research facility for studies of optical properties of glasses and other materials in infrared range (0.7 – 10 μm)

O.A. Prokhorenko
Laboratory of Glass Properties, 4 Sredny Prospekt, Research Bldg., 1st floor, St. Petersburg 190053 RUSSIA

Computer simulation of stages of glass manufacturing has a fair chance to become a powerful tool for optimization of melting, forming, annealing, and tempering operations. Modeling of heat transfer in molten glasses is an important part of computer simulation programs. One of the biggest problems is computation of radiative part of heat transfer. For successful solution of this problem correct mathematical description of transport of radiation and dependable experimental data on optical and thermal characteristics of all objects contributing in heat exchange by radiation are required. Absorption spectra of molten glasses, and emission spectra of ceramics and metals are the most important ones. In some cases data on reflection spectra of glasses and metals are needed. Taking into account necessity of computation of heat exchange in molten glasses containing inclusions (bubbles, seeds and particles) and at glass-refractory or glass-metal interfaces one should be able to study these compound subjects as well.

In 1994-1997 in the Laboratory of Glass Properties under support of the Center for Glass Research at Alfred University (Alfred, NY) development of method of measurement of absorption spectra of molten glasses had been performed. In 1998-2000 the devices and methods were improved in order to study glasses, which are subjects to irreversible changes at high temperatures. Real-time video monitoring of crystallization and bubbles formation in the course of registration of high-temperature spectra of the molten glass was applied at the first time.

In this article a new design of experimental facility, which allows studying different types of spectra of both semi-transparent and opaque materials, control and register different physico-chemical processes taking place in molten glasses and contact interfaces, is described.

Introduction

The purpose of this paper is to acquaint the specialists working in material science with our approach to experimental studies of optical properties in infrared range (absorption, emission, and reflection spectra, video monitoring). Dependable data on high-temperature optical properties of glasses and other materials (ceramics, metal, etc.) used at glass manufacturing are required for successful computer modeling of glass melting, forming, and annealing operations. In the present paper we tried to describe briefly experimental devices used for obtaining optical characteristics of glasses and melts essential for computation of radiative heat transfer. Some methods mentioned in this article were recently developed specially for the studies of different engineering materials playing important role in the heat exchange with molten glasses in the course of glass fusion and processing.

Absorption spectra

For computation of heat transfer within the system containing semi-transparent objects one needs experimental data on optical absorption of this object within infrared range regardless of mathematical approach used for thermaphysic calculations. Equation of transport of radiation (accurate solution) and approximation of diffusion of radiation require direct input of absorption spectra, whereas Rosseland and MRCA approximations of use values of λ_r (radiative conductivity) and ε (emissivity) determined from the spectra. Thus for determination of temperature distribution within the bulk of glass mass or glass articles during melting or forming one need to have its absorption spectra measured within a wide temperature range.

After a thorough study of publications on high-temperature absorption spectra of molten glasses we came to the conclusion that among the methods described in the literature sources none can be taken as a prototype for development of simple and dependable method. Our analysis of some results published in 1952 – 1997 is given in [1].

We have developed experimental devices and method, which allowed obtaining temperature dependence of infrared absorption spectra of glasses and melts within a broad temperature range (20 – 1500°C). This method based on step-by-step measurements of changes of absorption coefficients caused by the temperature increase is described in detail in [2].

General scheme of high-temperature spectrophotometer is presented in Fig. 1. High illumination of the device optical system allows measuring samples having optical densities **D** up to 3.0. Narrow-angle light beam transmitted through the sample chamber makes possible precise measurements of thick (up to 100 mm) specimens of highly transparent glasses [3].

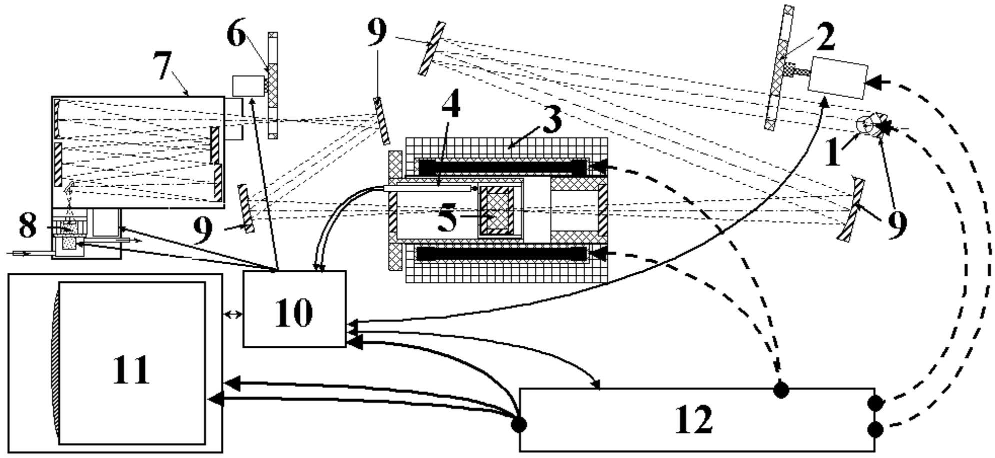

Fig. 1. General scheme of high-temperature spectrophotometer.
1 - light source; 2 - mechanical interrupter; 3 - furnace; 4 - thermocouple; 5 - cell; 6 - light filters turret; 7 - monochromator; 8 - detector cell; 9 - flat and parabolic mirrors; 10 - control unit; 11 - PC; 12 – stabilized power supply.

Using narrow-angle (single) beam optical scheme three devices (**SF-2.30-LGP**, **SF-3.30-LGP** and **SF-4.21-LGP**) were built. Recently the spectrophotometers were supplied with programmable all-in-one portable control units of a new generation (10) with built-in multidigit AD converters. Combination of advanced hardware and powerful software (all developed by LGP specialists) allowed setting complicated time-temperature regimes of registration of spectra. Selection of the regimes of measurements is made on the base of preliminary study of high-temperature behavior of the studied glass by using **spectrovision** technique (see below in the text). It helped to increase reproducibility of measurements of spectra of many molten glasses unstable at high temperatures (E-glasses, amber glasses, TV-funnel glasses).

Emission spectra

Unlike spectral emissivity of molten glasses this characteristic of opaque materials (metals, ceramics, etc.) can't be calculated from the absorption spectra. In order to measure emission spectra within the temperature range 500 - 1500°C devices **SF-3.30-LGP** and **SF-4.21-LGP** were supplied with additional parts (see Fig. 2). Mechanic interrupter (2a) and turret containing screen filters (6a) were positioned between the furnace (3) and the light filters turret (6). The interrupter (2a) made possible registration of radiation of the sample positioned inside the furnace by a detector cell (8) sensitive to impulse signal. The turret (6a) serves as an attenuator compensating sharp increase of level of radiation of the specimen caused by temperature increase. As the reference sample graphite cylinder mounted inside an argon-filled ceramic cell is used.

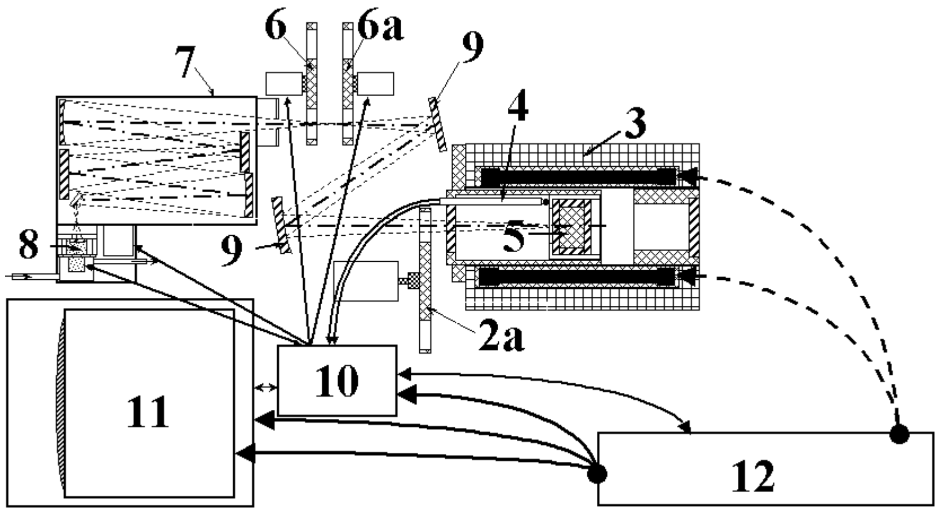

Fig. 2. Scheme of high-temperature device for measurements of emission spectra.
2a - mechanical interrupter; 3 - furnace; 4 - thermocouple; 5 - cell; 6 - light filters turret; 6a – screen filters turret; 7 - monochromator; 8 - detector cell; 9 - flat and parabolic mirrors; 10 - control unit; 11 - PC; 12 - stabilized power supply.

Reflection spectra

In many cases looses of energy connected with reflection of light from surface of a regular commercial glass can be taken into account using semi-empirical equations (see in [2]). Temperature and wavelength dependences of reflection coefficients of glasses coated with reflective films or anti-reflecting coatings, metals, and other materials should be measured. In Fig. 3 scheme of the device used for measurements of reflection spectra is presented. As the reference sample polished platinum plate is used.

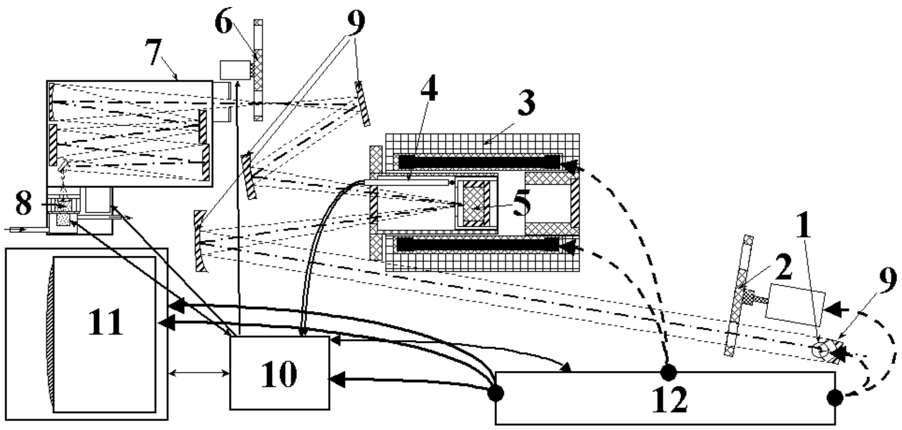

Fig. 3. Scheme of high-temperature device for measurements of reflection spectra.
1 – light source; 2 - mechanical interrupter; 3 - furnace; 4 - thermocouple; 5 - cell; 6 - light filters turret; 7 - monochromator; 8 - detector cell; 9 - flat and parabolic mirrors; 10 - control unit; 11 - PC; 12 – stabilized power supply.

Digital video monitoring

The optical absorption of the molten glasses is very sensitive to different irreversible processes taking place at the temperatures above ~1250ºC. Formation of bubbles and crystals in the bulk of the molten glass specimen or/and at melt-sapphire interfaces leads to sufficient increase of its optical absorption. Irreversible changes of red-ox state of the molten glasses containing different transition metals oxides can either decrease or increase their absorption. Irregular decrease or increase of optical absorption lead to gross errors of measurements of absorption spectra and determination of thermaphysical characteristics of molten glasses.

In order to make the process of registration of irreversible changes automatic and quantitative we supplied **SF-2.30-LGP** spectrophotometer with digital video monitoring system including CCD camera, video processor, and special imaging software. Detailed description of this system (**spectrovision**) and examples of its use can be found in [4]. The device, which allows simultaneous registration of high-temperature spectra and images of the studied specimen of the molten glass, is presented in Fig. 4.

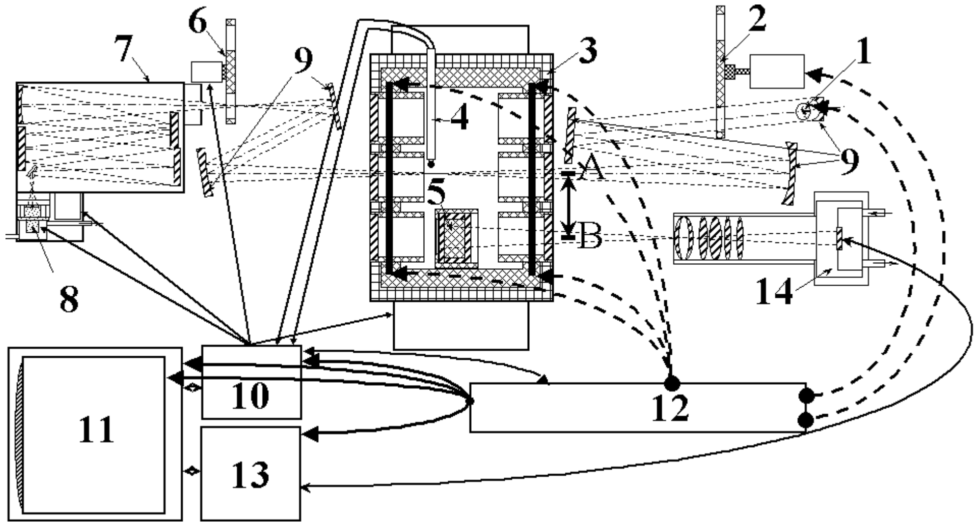

Fig. 4. Scheme of high-temperature spectrophotometer supplied with digital video monitoring system (spectrovision system).
1 – light source; 2 - mechanical interrupter; 3 - furnace; 4 - thermocouple; 5 - cell; 6 - light filters turret; 7 - monochromator; 8 - detector cell; 9 - flat and parabolic mirrors; 10 - control unit; 11 - PC; 12 – stabilized power supply; 13 – video processor; 14 – CCD camera.

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[1] O.A. Prokhorenko and O.V. Mazurin, Glass Phys. Chem. **25**, p. 159-162 (1999).

[2] O.A. Prokhorenko, O.V. Mazurin, M.V. Chistokolova, S.V. Tarakanov, Yu.E. Reznik and I.N. Anfimova, Glass Phys. Chem. **26**, p. 187-198 (2000).

[3] O.A. Prokhorenko in *ICG Annual meeting 2000 "Glass in the New Millennium, Challenges and Break-through Technologies",* Amsterdam (Netherlands) 2000, on CD, p. T3-6.

[4] O.A. Prokhorenko in *6th Internatioanal Conference "Advances in Fusion and Processing of Glass,* Ulm (Germany) 2000, p. 36-39.