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TEMPERATURE MEASUREMENT OF CERAMIC MATERIALS  
USING A MULTIWAVELENGTH PYROMETER

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Introduction

The surface temperatures of several pure ceramic materials (alumina, beryllia, magnesia, yttria and spinel) in the shape of pellets were measured using a multiwavelength pyrometer. In one of the measurements, radiation signal collection is provided simply by an optical fiber. In the other experiments, a 4.75 inch (12 cm) parabolic mirror collects the signal for the spectrometer. Temperature measurement using the traditional one- and two-color pyrometer for these ceramic materials is difficult because of their complex optical properties, such as low emissivity which varies with both temperature and wavelength. In at least one of the materials, yttria, the detected optical emission increased as the temperature was decreased due to such emissivity variation. The reasons for such changes are not known. The multiwavelength pyrometer has demonstrated its ability to measure surface temperatures under such conditions. Platinum electrodes were embedded in the ceramic pellets for resistance measurements as the temperature changed.

Method

The multiwavelength pyrometer consists of a spectrometer and a computer<sup>(1)</sup>. The light (signal) gathering function of the spectrometer is provided by a 3 meter (m) focal length parabolic mirror. At the operating field of view of 1 milli-radian, the radiation signal detected by the spectrometer comes from an area about 3 mm in diameter. By coupling an optical fiber to the spectrometer's wide angle attachment, which delivers radiation within its acceptance angle to the detector, the viewing angle of a 1.2 m long 750 μm diameter sapphire fiber is therefore also the spectrometer's field of view. The computer controls the spectrometer to acquire a spectrum, from which subsequent data analysis determines the temperature. To measure temperatures between 1,000 K and 2,000 K, the spectrometer was operated from 0.5 to 2.5 μm. The emitted/transmitted radiation is described by Planck's law of black body radiation<sup>(2)</sup>

$$L_{\lambda} = \epsilon_{\lambda} \tau_{\lambda} \frac{c_1}{\lambda^5} \frac{I}{\exp(c_2/\lambda T) - 1} = \epsilon_{\lambda} \tau_{\lambda} \frac{c_1}{\lambda^5} \exp(-c_2/\lambda T) \frac{I}{1 - \exp(-c_2/\lambda T)} \quad 1$$

where  $c_1, c_2$  are the radiation constants,  $L_{\lambda}$  is the radiation intensity,  $\epsilon_{\lambda}$  is the emissivity, and  $\tau_{\lambda}$  is the transmissivity of the optical medium (e.g. the optical fiber) between the pyrometer and the radiation source at wavelength  $\lambda$ .

For data analysis, Eqn. 1 is rewritten as

$$y = \left( \frac{\ln\left(\frac{c_1}{\lambda^5} \frac{1}{L_{\lambda}}\right)}{\frac{c_2}{\lambda}} \right) - \frac{\ln\left(1 - \exp\left(-\frac{c_2}{\lambda T}\right)\right)}{\frac{c_2}{\lambda}} = \frac{1}{T} - \frac{\lambda}{c_2} \ln(\tau_{\lambda}) \quad 2$$

This is the working equation of the traditional 1-color pyrometry method which requires knowing the emissivity and or transmissivity. For the multiwavelength pyrometer, neither quantity is required to determine temperature. Because the quantity  $(1 - \exp(-c_2/\lambda T))$  is practically unity at short wavelengths, its logarithm would be zero. We observe from Eqn. 2 that plotting the quantity  $y = \ln(c_1/(\lambda^5 L_{\lambda})) / (c_2/\lambda)$  as a function of  $\lambda$  would result in a straight line of slope  $\ln(\epsilon_{\lambda} \tau_{\lambda}) / c_2$  if  $\ln(\epsilon_{\lambda} \tau_{\lambda})$  is independent of wavelength. The quantity  $1/y$  at each wavelength  $\lambda$  is often referred to as the radiant temperature. The intercept of the straight line at  $\lambda=0$  is  $1/T$ , the reciprocal of the desired unknown temperature.

The resistance of the pellets was measured using a hand held digital multimeter capable of measuring resistance up to 20 mega ohms.

## Results

### (1) Spectra of alumina, beryllia, magnesia, yttria and spinel pellets recorded using a parabolic mirror

Alumina, beryllia, magnesia, yttria and spinel are common ceramic materials used in high temperature applications. All samples studied have a whitish granular appearance exhibiting very low emissivity, which may even be variable. Samples of these ceramics were prepared from pure commercial grade materials into pellet shapes about 1 cm in diameter, 5 mm thick. Platinum electrodes were embedded for resistivity measurements. These pellets were heated by a propane torch to elevated temperatures. The multiwavelength pyrometer was calibrated by focussing its parabolic mirror on a black body furnace ( $\epsilon=1$ ). The distances of the pyrometer from the black body furnace and from the measured samples were made equal. Spectra spanning the spectral region 0.5 to 2.5  $\mu\text{m}$  and possessing good signal to noise ratio were recorded in about 16 seconds at different propane torch flame intensities. They are shown in Figs. 1 to 5. These spectra were analyzed according to Eqn. 2. Some of the results are shown in Figs. 6 to 10. From the graph's intercept, the temperature of the measured surface was obtained. The emissivity of these materials over most of this spectral region is constant. In the case of yttria, the emissivity exhibited a marked irreversible change during the experiment.

### (2) Spectra of beryllia recorded using a sapphire optical fiber.

In one of the experiments, the signal gathering function of the parabolic mirror was replaced by a 1.2 m long, 750  $\mu\text{m}$  sapphire optical fiber. The normal calibration procedure using a black body furnace took into account the sapphire fiber's transmissivity. The fiber was inserted into the interior of a beryllia tube which has one of its ends closed. The end of the fiber was extended to within an inch of that end. The beryllia tube was part of the construction of a high temperature gas probe for measuring the gas temperature into which the probe was inserted. Measurement of the gas temperature inside the probe's protective beryllia tubing by a tungsten/rhenium thermocouple enabled the external gas temperature to be determined from a solution of the probe's heat transport equation. To prevent oxidation of the tungsten/rhenium thermocouples in the high temperature environment encountered inside the protective tubing, provision for an inert atmosphere such as argon was necessary. A measurement of the beryllia tubing's internal surface temperature using sapphire fiber optics would also lead to determination of the external gas temperature with the added advantage that it eliminates the burden to provide for an internal inert gas atmosphere demanded by the need to protect the chemical integrity of the tungsten/rhenium thermocouples.

The signal detected by the multiwavelength pyrometer using sapphire fiber input was weak but sufficient to determine the probe's internal surface temperature. The results are shown in Figs. 11 and 12.

### (3) Resistance measurement

At room temperature, the resistance of all the pellets was higher than the maximum measurable range (20 mega ohms) of the digital hand held multimeter that was used. As the temperature was raised by the propane torch flames, the resistance gradually decreased into the kilo ohm range. For two of the samples, beryllia and yttria, in order to make measurements at more than two temperatures, the propane torch's regulator knob was opened to different extent to produce flames of varying intensities. The temperatures and the corresponding resistances measured at the electrodes were thus obtained. Assuming that the ceramics were pure compounds containing no impurities, the measured electrical resistivity was due entirely to thermal excitation of intrinsic carriers into the conduction band.

The conductivity  $\sigma$  is therefore given by

$$\sigma = ne\mu \quad 3$$

where  $n$  is the carrier concentration at temperature  $T$ ,  $e$  is the electronic charge and  $\mu$  is the carrier mobility. Assuming Boltzman statistics to be valid at the experimental temperatures  $T$ ,  $n$  is given by

$$n = n_0 \exp\left(-\frac{E}{kT}\right) \quad 4$$

with  $k$  being the Boltzman constant,  $n_0$  the intrinsic carrier concentration,  $E$  is the activation energy. The reciprocal of its conductivity is the resistivity  $\rho$ , which is given by

$$\rho = \rho_0 \exp\left(\frac{E}{kT}\right) \quad 5$$

A plot of the logarithm of the measured resistance vs the reciprocal of the temperature is a straight line of slope  $E/k$ .

The analyses of beryllia and yttria data are shown in Figs. 13 and 14. Their activation energies are determined to be 3.05 and 1.9 eV. The magnitude of activation in a semi-conductor is a measure of the shallowness or depth of its carrier (dopant) from the conduction or valence bands. These large activation energies are indications that the pellet materials are very good insulators at low temperature. These are just commercial grade (pure) ceramic substances, there could be impurity dopants. The carriers are therefore a combination of them and those produced by pure thermal excitation at high enough temperatures. For an individual pellet, the measured resistance between electrodes can be calibrated to provide rough temperature measurements. The changes in resistance collaborate the pyrometer temperatures closely.

### Discussion

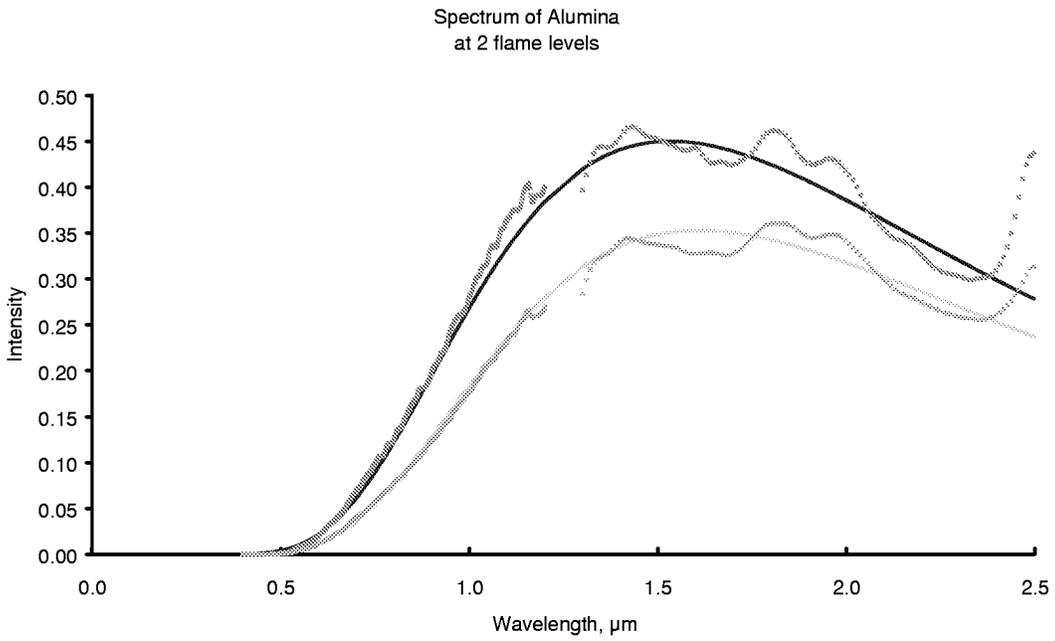
Temperature measurement of ceramic materials using pyrometers is known to be difficult. All the materials studied have low emissivity. In the analyses shown in Figs. 6 to 10, the regions of constant emissivity in the spectral regions are obvious. Constant emissivity in a spectral region allows one to measure temperatures easily. The propane torch can raise the ceramic materials to temperatures ranging from 1700 K to 1900 K. The ceramic emissivities are in the neighborhood of 0.1 to 0.3. In the case of yttria, it was observed that the intensity of radiation emitted by its surface increased as the intensity of the flame was lowered when the propane gas throttle valve was reduced. Temperature and time history variation of emissivity is known to be exhibited by thermal barrier coating (TBC) zirconia ceramics stabilized with yttria. Analysis of the yttria spectra at the two heating levels (Fig. 10) yielded two temperatures and emissivities. Traditional 1- or 2-color pyrometers would be unable to make temperature measurements in this situation. Examination of the yttria surface afterwards revealed that it had changed from the initial whitish yellow color to a grey slightly blackish coloration. During the time that the experiment was changed from one heating level to the next, a structural or chemical transformation must have occurred that resulted in changes in the material's emissivity. In the series of experiments that investigated the changes of the yttria sample's resistance with temperatures, a different area of the sample still possessing the whitish yellow coloration was focused to collect pyrometer data for temperature determination. No further change in emissivity was observed.

### Conclusion

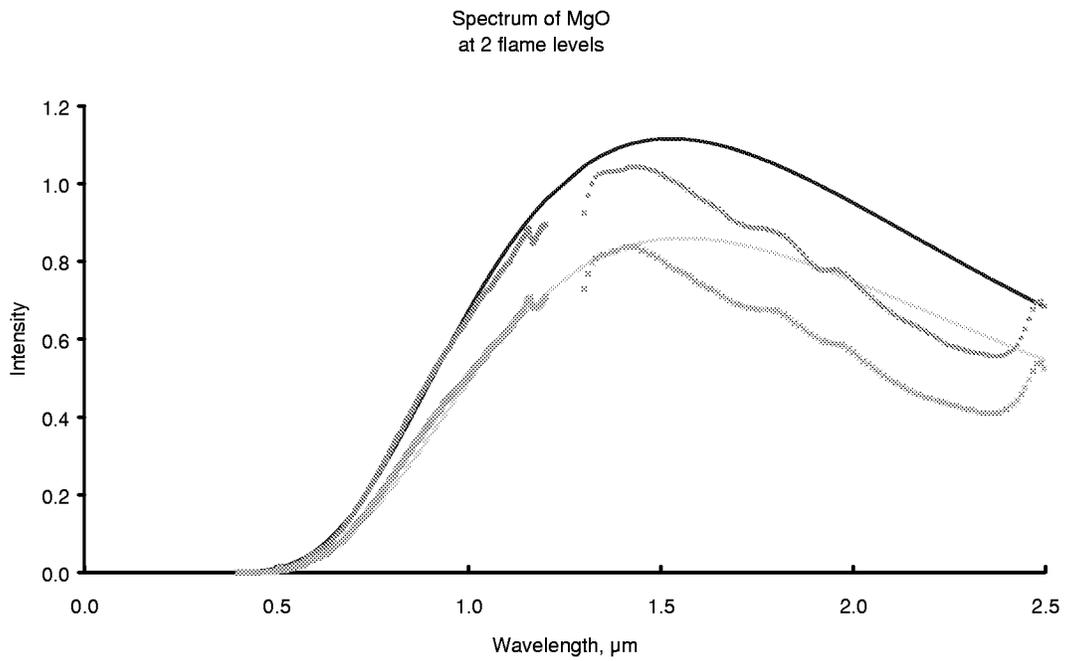
The surface temperatures of several ceramic materials were measured using the multiwavelength pyrometer. The multiwavelength pyrometer determined temperature by transforming the radiation spectrum over a broad wavelength region to produce a straight line which intercepted the graphical axis. The inverse of the intercept gives the temperature. A minimum of two wavelengths spanning the wavelength regions would suffice because two points would determine a straight line to produce a slope and an intercept. However confidence that the emissivity in the spectral region under consideration is constant can only be provided by a redundancy of data. Replacement of the sapphire optical fiber by an almost non-absorbing silica fiber viewing through an aperture arrangement which reduces the fiber's field of view will increase the detectable optical signal hundreds of fold, thus enhancing both accuracy and data acquisition speed. Temperature measurement would be difficult by traditional pyrometers on substances such as yttria which exhibits variable emissivity. High activation energy, characteristic of insulating ceramic materials is determined for beryllia and yttria.

### References

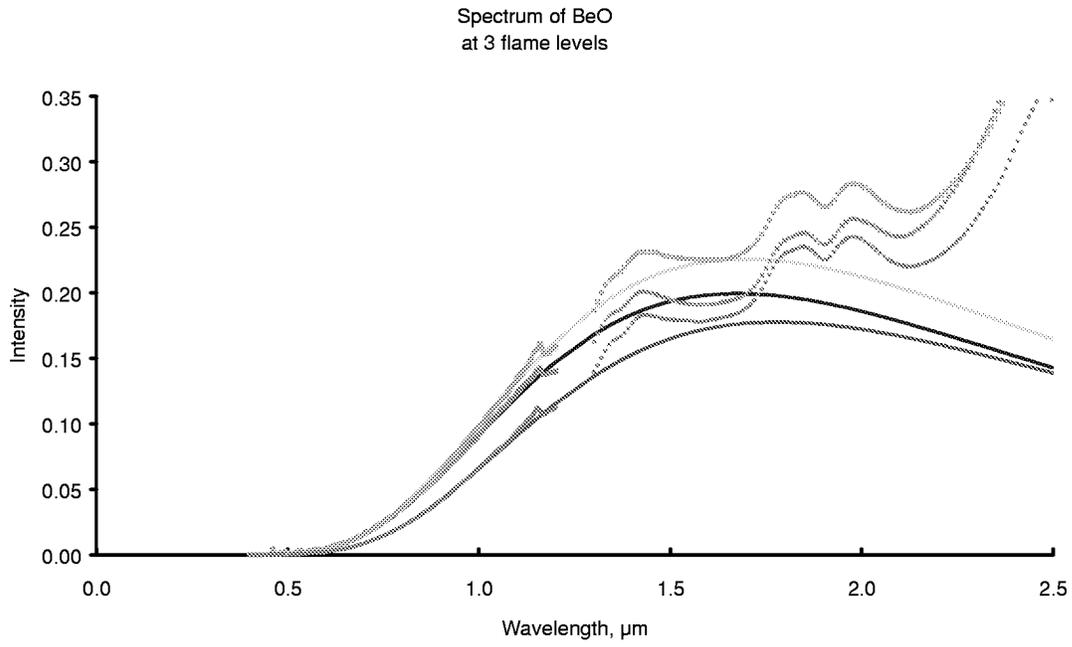
1. Ng, D., Temperature Measurement of a Glass Material Using a Multiwavelength Pyrometer, NASA TM 107433, April 1997.
2. DeWitt, D.P., Nutter, G.D., Theory and Practice of Radiation Thermometry, John Wiley & Sons, New York, 1988.



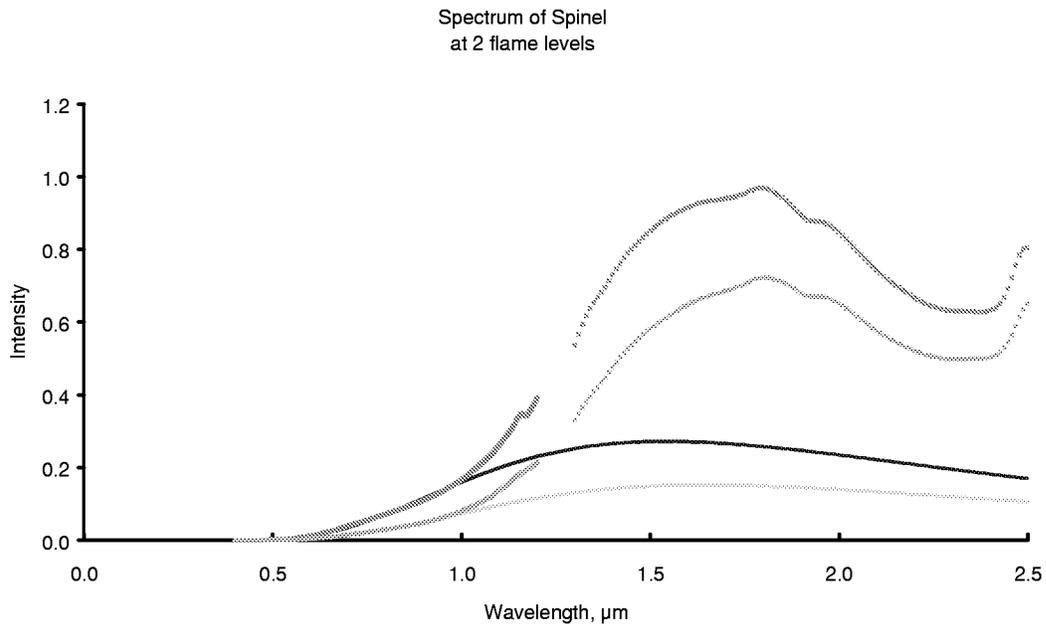
**Figure 1** Alumina spectra



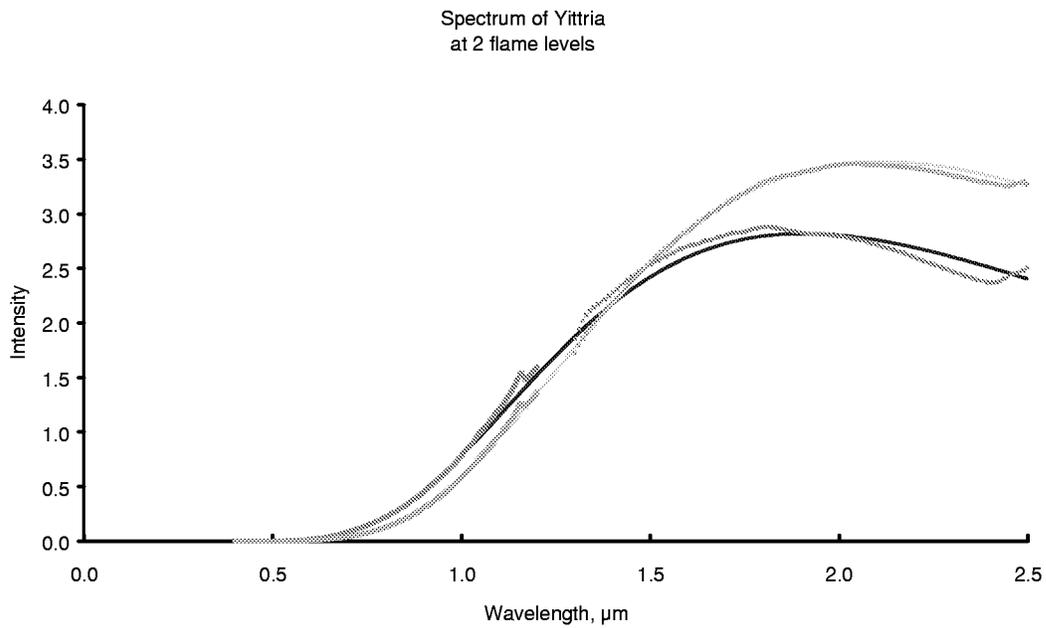
**Figure 2** MgO spectra



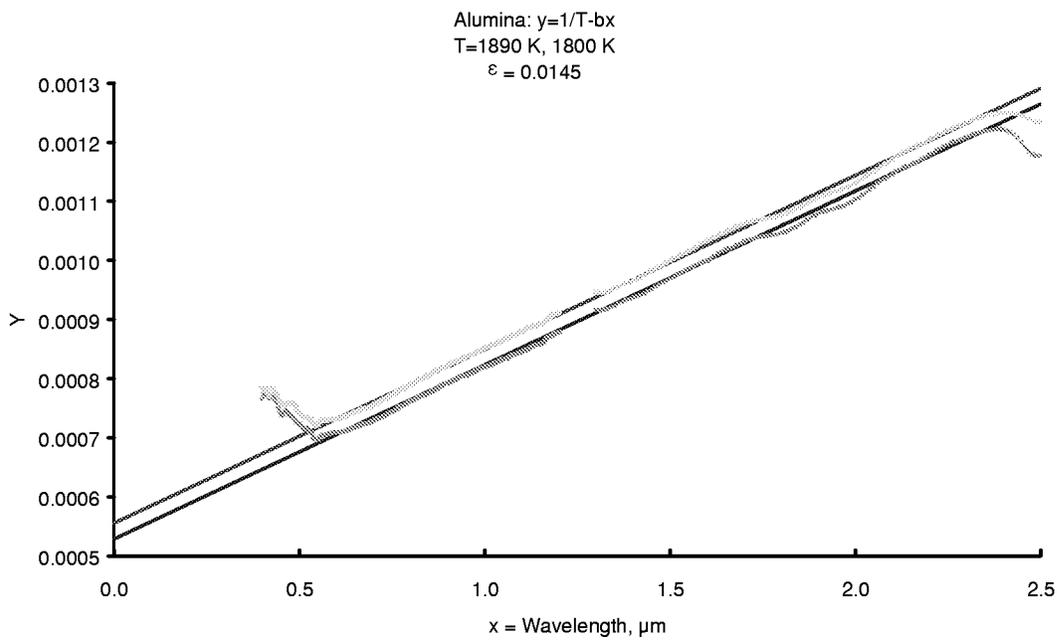
**Figure 3** BeO spectra



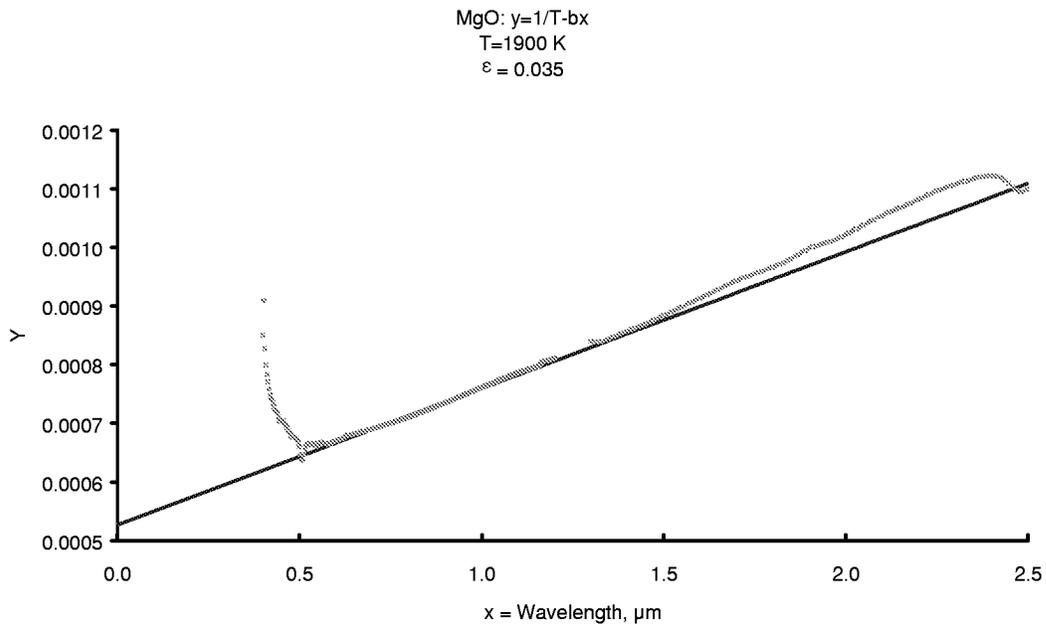
**Figure 4** Spinel spectra



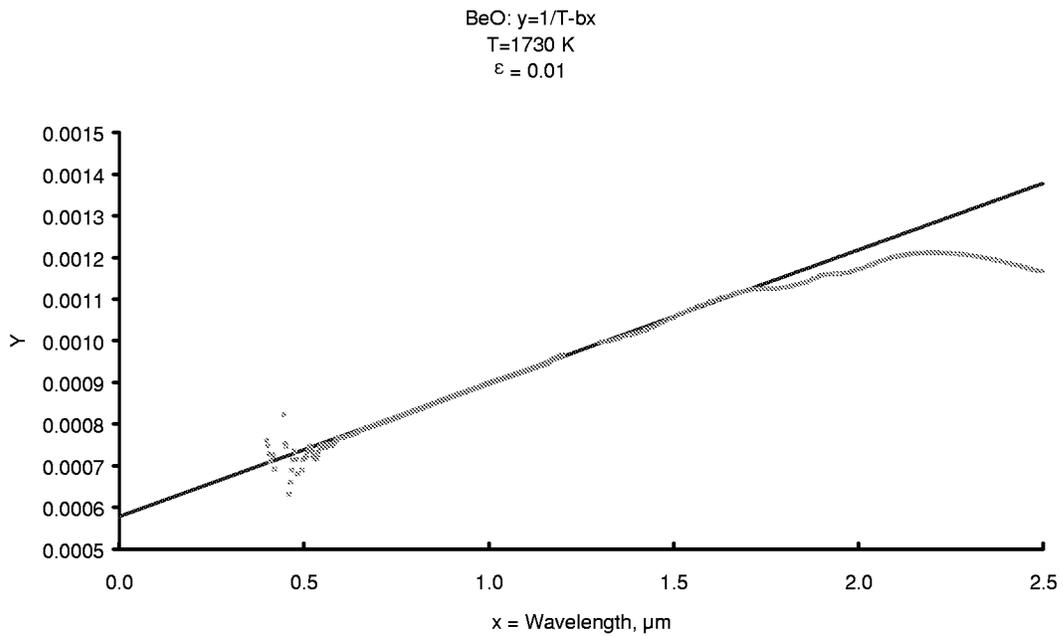
**Figure 5**      Yttria spectra



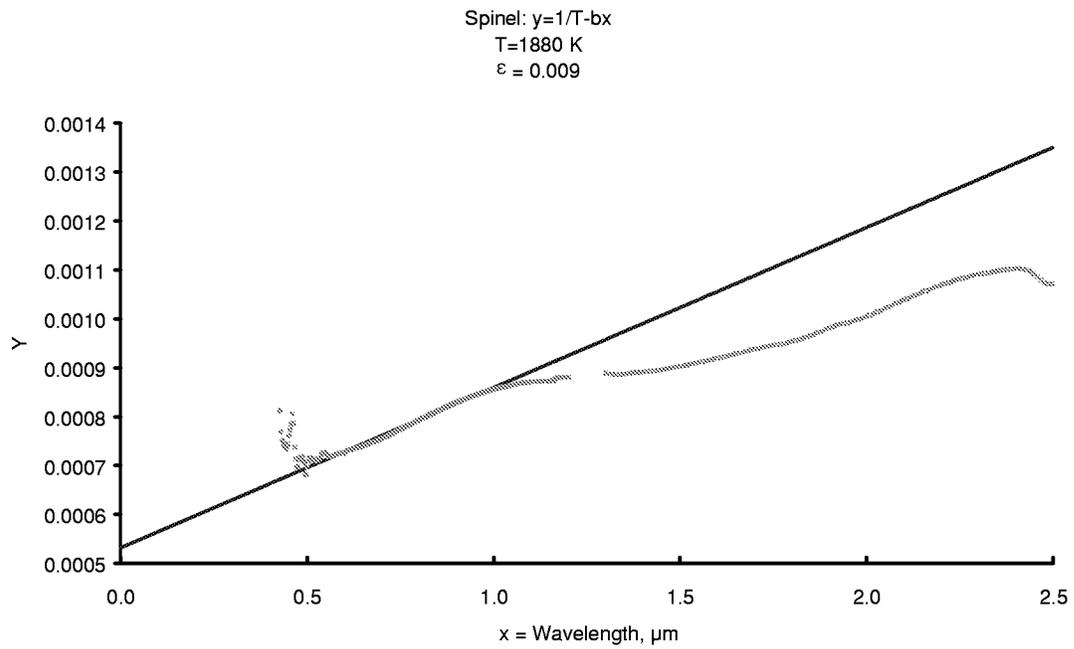
**Figure 6**      Analysis of alumina spectra



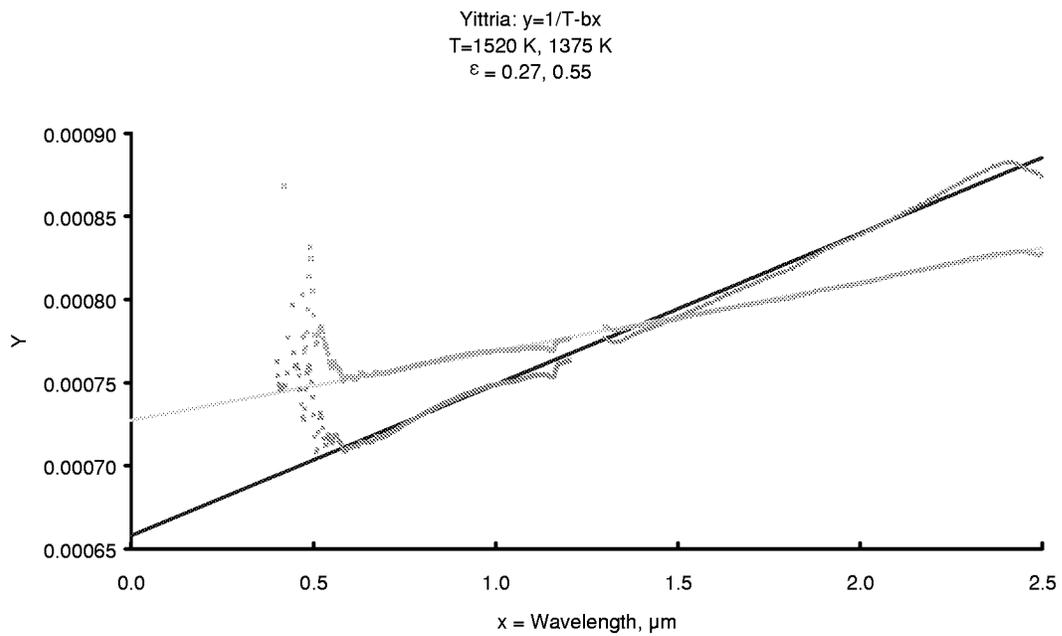
**Figure 7** Analysis of MgO spectra



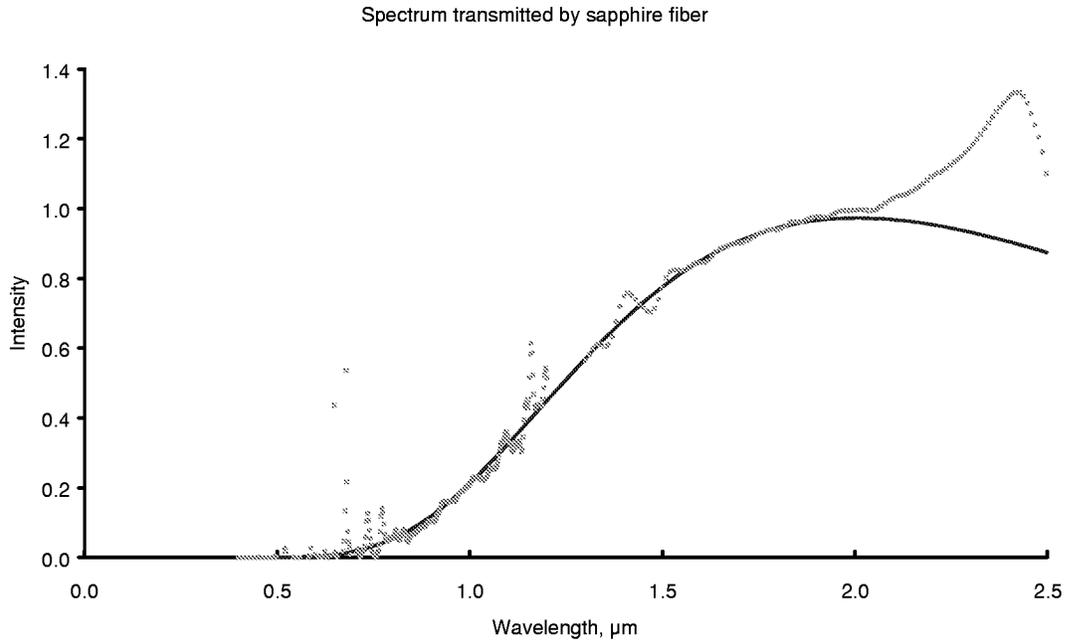
**Figure 8** Analysis of BeO spectra



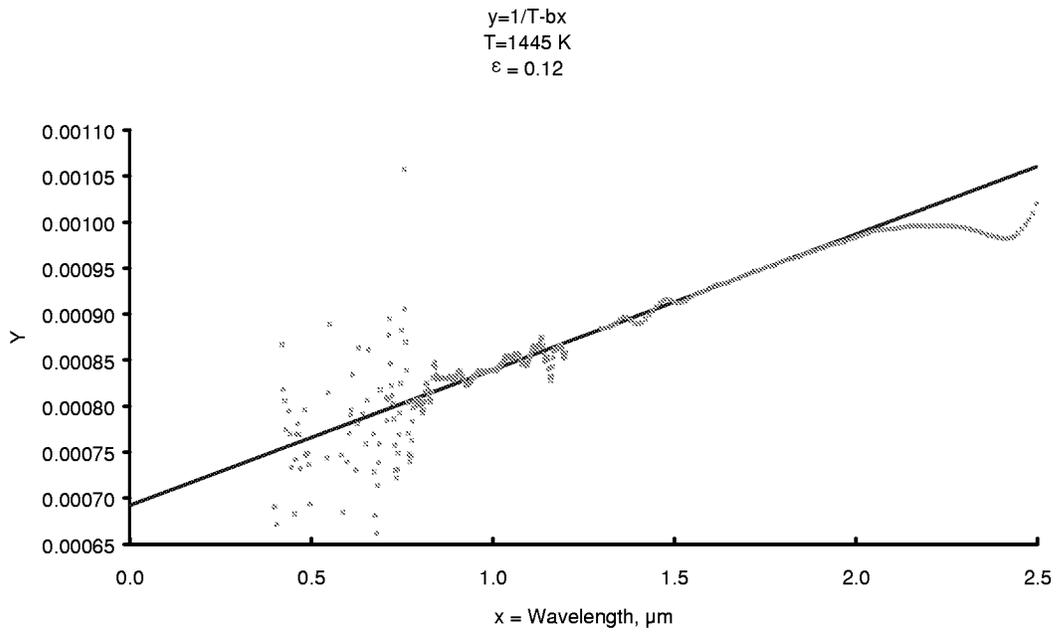
**Figure 9** Analysis of spinel spectra



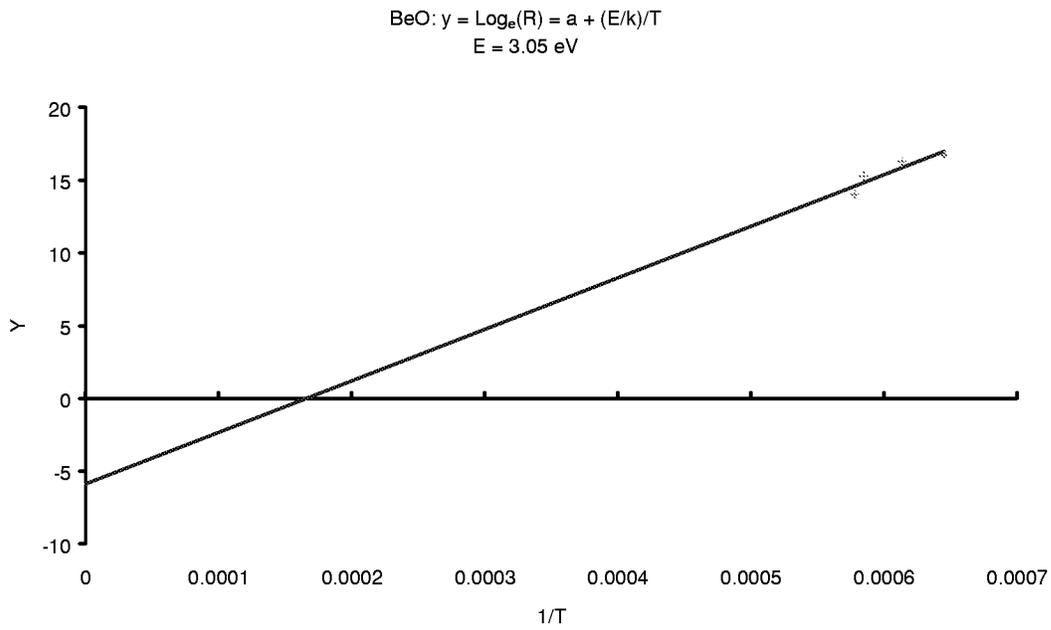
**Figure 10** Analysis of yttria spectra



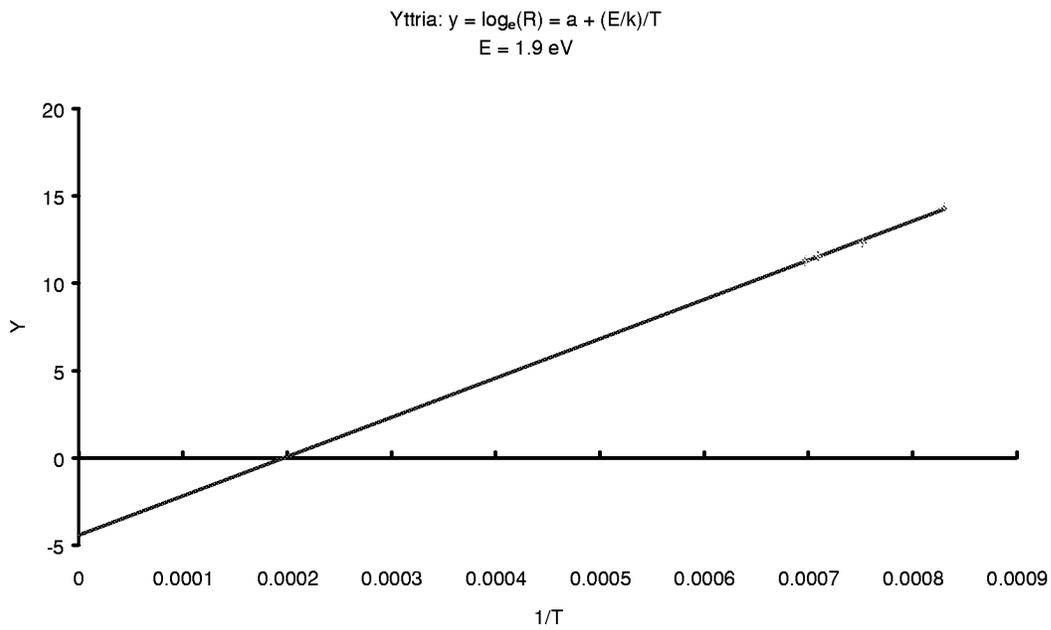
**Figure 11** Sapphire fiber collected BeO spectra



**Figure 12** Analysis of apphire fiber collected BeO spectra



**Figure 13** Resistance vs 1/T plot of BeO



**Figure 14** Resistance vs 1/T plot of yttria

# REPORT DOCUMENTATION PAGE

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