Measuring absorptivity of ceramic materials at high temperatures

in Gyrotron Ceramics Sintering System

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The development of processes based on the microwave heating of materials requires knowledge of the dielectric properties of the materials and their temperature dependences. In this work we measure the microwave radiation absorption coefficient in alumina-based ceramic materials over a wide range of temperatures. The method is based on comparing the Q-factors of an unloaded resonator and a resonator with an absorbing sample placed inside [1], [2]. In our experiments the working chamber of the gyrotron complex is in fact a multimode ($L \gg \lambda$) untuned resonator [3]. A distinctive feature of this method is the use of radiation simultaneously for both diagnosis of properties (measurement of the absorption coefficient) and heating of the sample.

In the experiments we investigated absorptivity of two types of alumina ceramics materials: A0 (> 99.5% Al_2O_3) and AZ11 (88% Al_2O_3 + 11% ZrO_2 + 1% Y_2O_3). Samples of both compositions were pre-sintered in a resistive heating furnace to a density of about 80% of the theoretical density (the porosity was near 20%). Basing on the measured absorptivity and theoretical calculations of the absorption coefficients of the samples under conditions of omnidirectional microwave irradiation and the dielectric characteristics of materials of complex composition, the loss tangents of the studied materials were determined.

The Q-factor of the resonator was measured using a microwave power meter connected to the resonator through a small cross-section aperture. The measured power P is proportional to the intensity of the electromagnetic field inside the resonator. Comparing the values of the power meter signals in cases of an unloaded resonator and a resonator loaded with a sample, it is possible to determine the ratio of the Q-factors in these two cases (the index 0 refers to the unloaded resonator)

$$\frac{Q}{Q_0} = \frac{P}{P_0}. \tag{1}$$

Using the relation (1) and determining from independent measurements the Q-factor of the unloaded resonator Q_0, we obtained the expression for the absorption coefficient α of microwave radiation in the sample

$$\alpha = \frac{\omega V}{c S Q_0}\left(\frac{P_0}{P} - 1\right), \tag{2}$$

where $\omega = 2\pi f$, f=24 GHz is the frequency of the electromagnetic field, c is the speed of light, V is the volume of the chamber, S is the sample surface area.

By processing the power meter data, we obtain an experimental dependence of the absorption coefficient on temperature. Knowing the values of the microwave radiation absorption coefficient in the sample and the real part of the dielectric permittivity of the sample, we can calculate the loss tangent.

Shown in Fig. 1 is one of the realization of the heating of A0 sample.
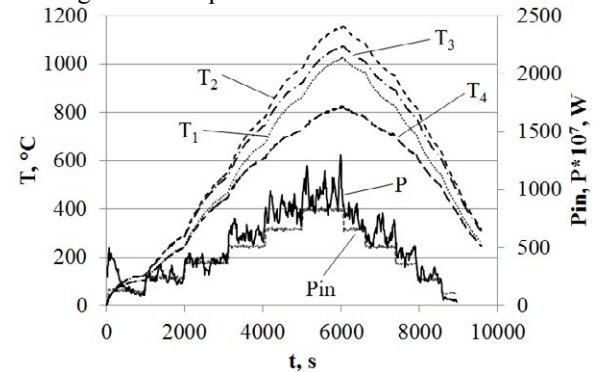
Fig. 1. The experimental realization of the heating of the A0 sample: T_1, T_2, T_3, T_4 are the temperatures at various points in the sample, P_{in} is the microwave power delivered to the working chamber, P is the power measured by the power meter

For the theoretical calculation of the absorption coefficient, we used the well-known solution to the problem of absorption of electromagnetic radiation in a plane dielectric layer of finite thickness d [2], [4]. The thickness of the samples was much smaller than their transversal dimensions, so the contribution of the side surfaces was not taken into account. The absorption coefficient α in the material was calculated according to the formula:

$$\alpha = \int_0^{\frac{\pi}{2}} A(\theta, \varepsilon', \varepsilon'', d, \omega) \sin\theta \cos\theta \, d\theta, \tag{3}$$

where $A = \frac{A_{TE} + A_{TM}}{2}$ is the average absorption coefficient, A_{TE} and A_{TM} are the absorption coefficients for TE and TM incident waves correspondingly, θ is the angle between the incident wave and the surface of the sample, ε' и ε'' are the real and imaginary parts of the effective dielectric permittivity of the sample, respectively. To calculate the effective dielectric permittivity of a sample with complicated compositions including pores, we used the effective medium approximation (Bruggeman's model) [5]. The dielectric permittivity values of Al_2O_3 and ZrO_2 at room temperature were taken from [6], [7]. In the temperature range of these experiments, the real parts of the die-

lectric permittivity of these oxides in the centimeter wavelength range have weak dependence on the temperature [8]. This allowed us to consider the real parts of effective dielectric permittivity as a constant value.

We can determine the temperature dependence of the loss tangent ($\tan \delta = \varepsilon'' / \varepsilon'$) using the theoretical dependence $\alpha(\tan \delta)$ (3) and the experimental temperature dependence of the absorption coefficient α. The results obtained are shown in Fig. 2. Here, data on the loss tangent for alumina ceramics of similar composition (99.5 – 99.7% Al_2O_3) at a frequency of 15 GHz at room temperature [9] are given for comparison.
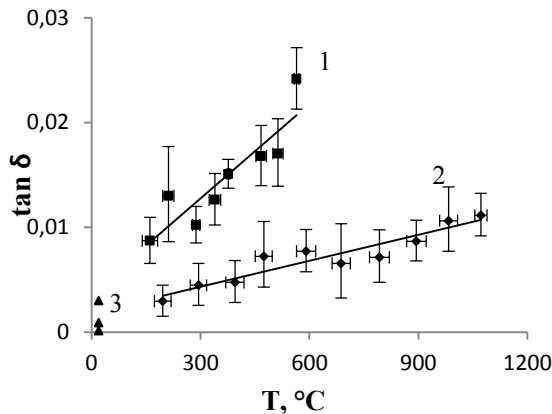
Fig. 2. Dependence of the loss tangent on temperature: 1 – AZ11, 2 – A0, 3 – alumina ceramics (99.5 - 99.7% Al_2O_3) according to [9] at f = 15 GHz

Conclusions

We obtained the temperature dependence of the loss tangent in ceramic materials of complex composition. The method is based on a comparison of the measured intensity of electromagnetic radiation in the working chamber of a gyrotron complex with and without a sample inside the chamber. The method makes it possible to carry out measurements at high material temperatures, since it does not require contact of the measuring elements with hot surfaces.

The measurements were carried out using the frequency typically used for microwave processing.

The accuracy of determining the absorption coefficients is 10-30%, which is acceptable for most problems associated with the use of microwave heating for research and development of such applied processes as sintering and joining of ceramic and composite materials.

It is shown that for known dielectric constants of the components of the measured composite materials, it is possible to determine the loss tangent as a function of the material temperature.

The following restrictions apply to the use of this method:

1) dimensions of the sample must be much greater than the wavelength of the radiation,

2) characteristic transverse dimensions of the sample must be much greater than its thickness, which is necessary for the correct use of the model of a uniformly heated plane infinite dielectric layer,

3) temperature of the sample is determined by the incident power and the exposure time, and therefore is not an independent parameter in the measurements.

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