

THERMAL CHARACTERISATION OF SINTERED MAGNESIA-ALUMINA BY THE PHOTOACOUSTIC TECHNIQUE

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ABSTRACT

The photoacoustic effect was used in this study to thermally characterise ceramic materials with refractory properties, particularly the magnesia-alumina ($MgAl_2O_4$) spinel. The usefulness of the methods involving the photoacoustic effect and thermal relaxation was shown. The measurement data exhibited good accuracy when compared with data obtained by other experimental methods.

1. INTRODUCTION

Materials thermal properties are of great importance in their basic aspects as well as from the point of view of their applications. Thermal characterisation is very sensitive to material structure and serves as a guide to the choice and/or preparation parameters of the material at issue in the manufacture of a large number of items, e.g. in the automobile, refractories, steel, ceramic industry, etc.

2. PHOTOACOUSTIC EFFECT

The photoacoustic effect consists of producing sound when light is made to impinge periodically on a sample in a closed chamber. Light absorption produces a periodic heating that is diffused through the sample. As a result, a layer of air forms at the sample-gas boundary, which periodically expands and contracts giving rise to pressure fluctuations. This movement generates a photoacoustic signal in the chamber containing the sample. A microphone detects the arising sound.

3. EXPERIMENTAL

The analysed samples were obtained by artificial sintering of the spinel ceramic material. The reaction route consisted of mixing magnesium carbonate ($MgCO_3$) and alumina (Al_2O_3), with calcium carbonate ($CaCO_3$) as a flux, for different quantities of reagents. The foregoing reaction was produced in a convection kiln with electrical resistance charge and discharge and a microwave kiln in which a crucible system designed for this purpose was used. Samples were taken of sintered material from different areas of the product, which were mechanically polished for the thermal characterisation. The photoacoustic technique, unlike other methods, requires very small samples about 300 microns thick, measuring around 0.5×0.5 cm. It is thus possible to optimise the obtainment of samples with appropriate thermal properties, by making a point measurement.

SAMPLES	PHASE	wt%	Thickness μm	$\alpha(cm^2/s)$	$\rho C(J/cm^3 \cdot K)$
1R	$MgAl_2O_4$	89	295	0.056 ± 0.014	8.29 ± 1.28
	MgO	10			
2M	$MgAl_2O_4$	86	290	0.046 ± 0.015	6.21 ± 0.60
	$Ca_3Al_2O_6$	4			
	$CaAl_2O_4$	8			

Table 1. Results of the thermal measurements performed on the sintered material samples containing the magnesia-alumina spinel as major phase.

The thermal diffusivity measurement technique (photoacoustic technique) was also applied to samples with pure ceramic materials components, always using ceramic materials. The measurements are set out in Table 2. Table 3 presents the experimental data obtained by other authors with other experimental methods [Y. S. Touloukian, R. W. Powell, C. Y. Ho and M. C. Nicolaou, Thermophysical Properties of Matter, Plenum, Vol. 10, New York, 1970].

Sample	Thickness μm	$\alpha (cm^2/s)$
MgO	346	0.179 ± 0.010
$CaCO_3$	197	0.0093 ± 0.001
Al_2O_3	255	0.075 ± 0.004

Table 2. Thermal diffusivity measurements of samples of pure components by the photoacoustic technique using the open cell mode.

Sample	$\alpha (cm^2/s)$	Temperature K
Al_2O_3	0.0687	293
MgO	0.190	272
$CaCO_3$	0.00156	318
$MgAl_2O_4$	0.0341	298

Table 3. Thermal diffusivity data obtained using a different technique from the photoacoustic technique.

4. CONCLUSIONS

The study of ceramic spinels performed in this work evidences the usefulness of the photoacoustic effect and thermal relaxation in determining the thermal properties of ceramic materials with refractory properties. The techniques mentioned, unlike other methods, require very small samples with a thickness of around 300 microns, measuring about 0.5 x 0.5 cm. This feature allows thermal properties to be determined in terms of the position in a bar or crucible. It is thus possible to optimise the obtainment of samples with adequate thermal properties.

The samples, particularly the magnesia-alumina samples which were thermally characterised, whether they were prepared by resistive heating or in a microwave kiln, exhibited very similar thermal diffusivity values, while the results of the heating capacity measurement per unit volume presented a greater scatter. This last effect was due to the heating medium. The samples prepared in the microwave kiln achieved poor sintering compared to the ones sintered in the kiln fitted with resistances. The thermal diffusivity data exhibited good accuracy on comparing these with data found by other experimental methods.