

The Microwave Measurements of the Dielectric Properties of Ceramics at High Temperature

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ABSTRACT. The girl's section in King Abdulaziz University has got a good chance that the Saudi girl can get to Postgraduate research in her country via the joint supervision program between King Abdulaziz University and British Universities. It is a convenient program to get to the Ph.D. especially in the College of Science that the students themselves built the modern laboratories with the oversight of British supervisors.

Ceramics are important materials for modern industries especially when processed by the microwave energy. To control this process the dielectric properties and permittivity of ceramic materials at high temperature and in the microwave range must be known.

A microwave measurements system of the cavity perturbation technique has been designed and built to measure the dielectric properties of any kind of materials whether solid, liquid or powder materials. This system is fully computer automated and it has the capability of accurately measuring the complex permittivity of a very low loss factor, less than 0.0001 of the quality factor, $Q = 10000$, at high temperature range of 25°C to 200°C and frequency range of 0.6 to 3.8 GHz.

Introduction

Research indicates that ceramics are impertinent materials for modern industries. They are inorganic materials of wide usage particularly when used as the elements of microwave microcircuits^[1]. Fine ceramics, which are excellent in resistance to heat and corrosion, are promising as structural materials capable of replacing metal under high temperature use^[2]. One of the most important

properties of ceramic materials is that they retain their structural properties at high temperatures; they usually present a high melting point and this property hinders the processing of this kind of material under conventional heat^[3].

Microwave energy offers many advantages over conventional heating. These advantages present new and unique opportunities to process materials with greater flexibility, greater speed, and time and energy saving. Microwave energy appears to be especially advantageous for the processing of many types of ceramics requiring high temperatures. This is due to the increased coupling efficiency of ceramics with microwaves as they are heated and to the more uniform, volumetric, heating provided by microwaves^[4,5]. In spite of the great potential of microwave energy in the field of ceramics processing, there are some barriers that must be overcome before its widespread use will be realized. Thermal runaway, the sudden rise of temperature, which causes material damage during the microwave heating, is one of the challenges in microwave heating which faced the growing of the microwave processing of ceramics^[6]. Microwave heating is an energy transfer that the material will convert the microwave energy into heat within itself through the process of dipole rotation or ionic conduction. Conversion microwave energy into heat inside the material is the mechanism of heating which depends on the nature of the material; its electronic, molecular, crystalline structure, its microstructure, and impurity content^[7]. These processes are electronic and depend on the dielectric properties of the material which means that to control the microwave processing of ceramics the dielectric properties of these materials must be known^[8].

Dielectric constant or the complex permittivity real and imaginary parts, ϵ' and ϵ'' , are parameters that describe the behaviour of a dielectric material under the influence of a microwave field. They both affect the power absorbed and the half-power depth, they describe how microwaves penetrate and propagate through an absorbing material, and they influence the volumetric heating of a given material^[9].

Techniques of microwave measurements of the complex permittivity at high temperature is the most difficult part in microwave processing of ceramics and it can be classified as; free space, transmission line, and cavity perturbation. Cavity perturbation technique is the best method for these measurements. It is distinguished by its higher measuring precision and simple calculations and does not have a special requirement for one geometry, size and kind of the sample such as solid, powder, and liquid^[10].

The third system in the world and the first or the unique system in the Middle East of the microwave measurement of the dielectric properties of ceramics has been built in King Abdulaziz University using the cavity perturbation technique

in frequency range 615-3800 MHz and the temperature range 25-2000°C. CMS or the Computerized Measurement System is a software scheme has been done individually. It monitors the attached measurement devices and controls the moving devices of the system. CMS capable of estimating, finding, and measuring the resonant frequency f and the loaded Q-factor Q of the cavity with and without the sample. It can calculate the two parts of the complex permittivity, ϵ' and ϵ'' , using f and Q . Also, QMS display the change of ϵ' and ϵ'' versus the change of temperature in several formats such as graphs, tables, and separate sheets. Finally, it can send these sheets to the Excel for analysis.

Device Construction

The main components of this system shown in Fig. (1) are:

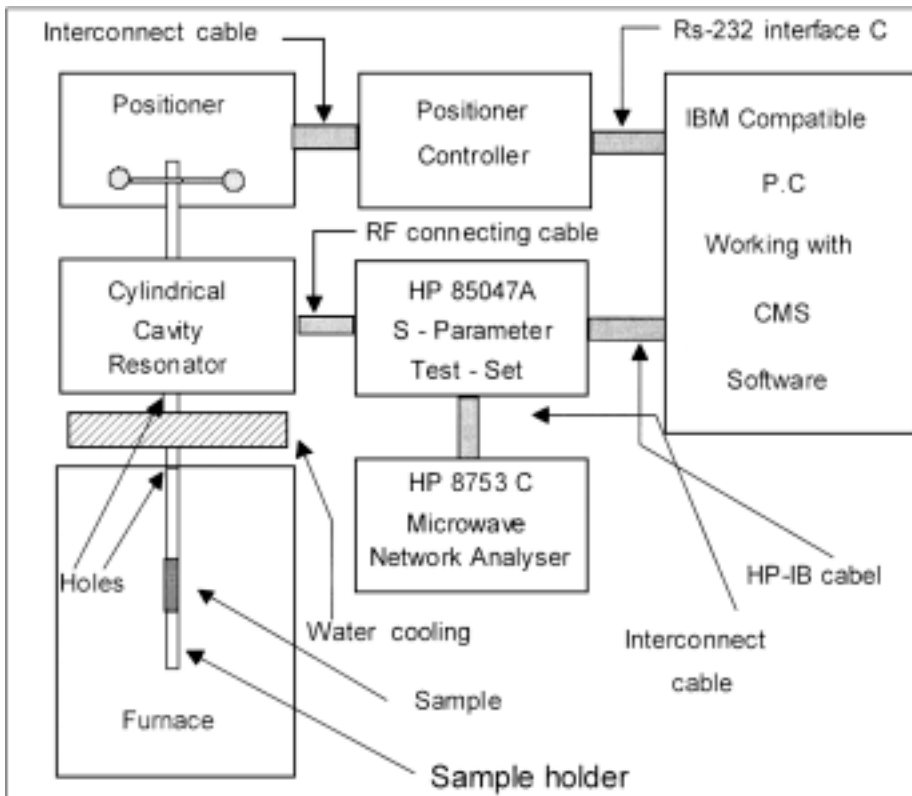


FIG. 1. The measurement system.

- A curricular cylindrical resonator cavity of suitable dimensions to resonate at the desired frequencies. It is made of copper with dimensions of 373 mm and 37.3 mm in diameter and length respectively. A schematic diagram of the cavity dimensions is given in Fig. (2).

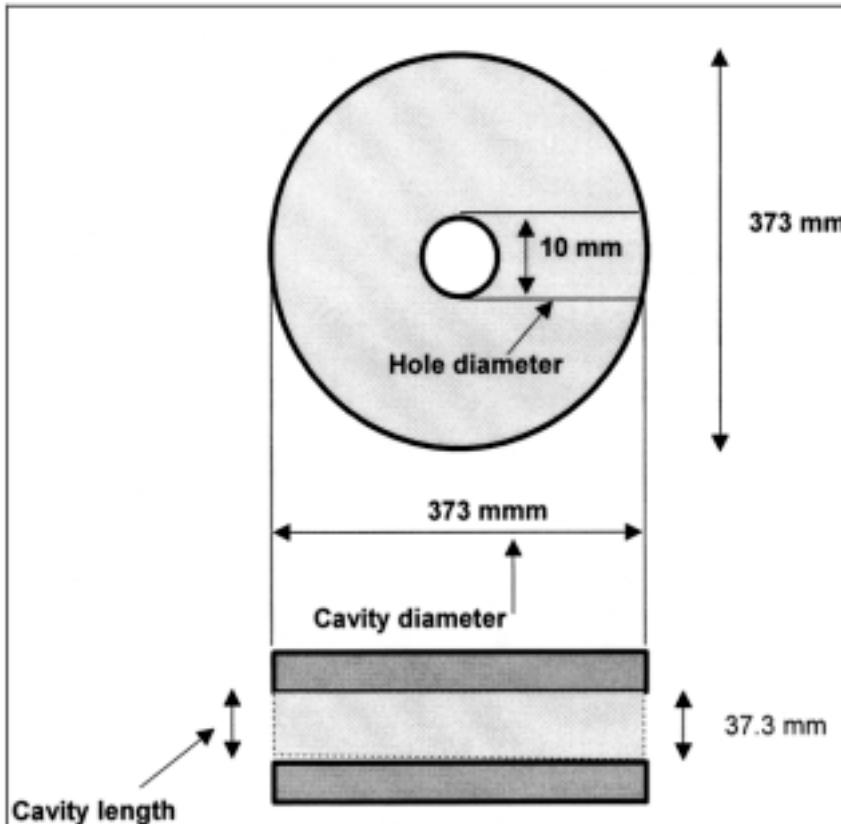


FIG. 2. Cavity dimensions.

Figure 2 shows that there are holes in the two end plates of the cavity, 10 mm in diameter. These holes were made to simplify the movement of the sample between the cavity and the furnace an expected variation in the electric field distribution inside the cavity, due to these holes, can be neglected when comparing the internal diameter of the cavity with the holes' diameters as shown in Fig. (2).

- A BLF model is a conventional chamber furnace with an electrically operated elevator hearth. The maximum operating temperature is 1800°C (Temperature control by Eurotherms 902P programmer). A contactor isolates the elements in the event of an over temperature condition, and also when the hearth is fully lowered. Cooling fans provide forced air cooling of the case.

- A positioner which is used to position the sample inside the cavity or inside the furnace is single axis Unidex 1 Microstepping Driver is a microprocessor-based motion which can control the most applicable, low and high power programmable positioning. It is linked to the PC via its port A, using RS 232 cable.

- A Hewlett Packard 8753C vector network analyser, VNA, with HP 85047A S-Parameter Test-Set and sweeping frequencies kHz to 6 GHz is a two-port device which can sweep and transfer a sine wave signal to stimulate the device under test, the cavity, and detect the reflected signal. Also it measures the separated signals, from the desired signal ratios, and display the results. The computer interfaces with VNA using an IEEE488 interface with the HP 82335B HP.DLL for windows and DOS.

- S-parameter can define the ratios of the reflected and transmitted travelling waves measured at the network ports. The two-port device is modelled with S-parameter in Fig. (3).

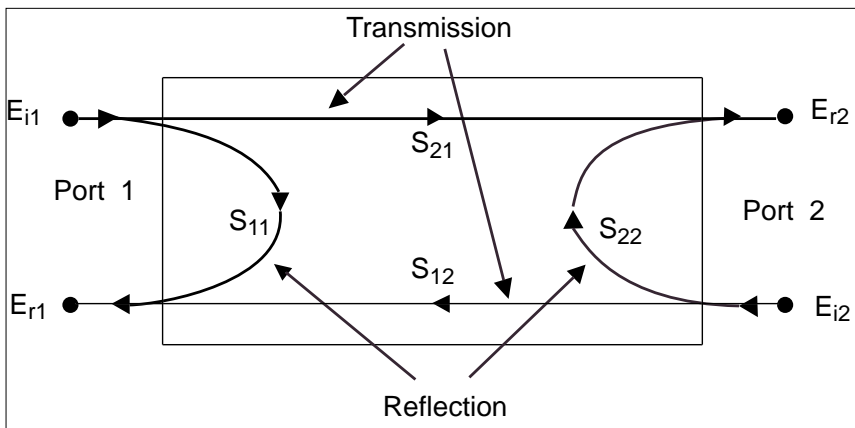


FIG. 3. S-parameter model for a two-port linear network.

- A silica tube, is used to hold the sample during the measurements. It was made particularly for this measurements system so that it can tolerate the high temperature, about (2000°C).

- A Mettler Toledo B balance lines College-B/C B/GB it is a very sensitive balance used to obtain accurate weighing results. It is employed to weigh the sample for the density determination.

The Computerized Measurement System

CMS, is based on a piece of software, Visual Basic application, which is used to control the system to measure the dielectric properties of any kind of materials such as powder, solid, and liquid, at low and high temperatures. It monitors the attached measurement device, VNA, and controls the moving device, the positioner, of the measurement system via the interfaces, (RS-232 C/dais chain) and (HP 82335 HP-IB) respectively.

The measurement methods of this technique is based on perturbation technique where the real, ϵ' , and the imaginary, ϵ'' , parts of the complex permittivity, in term of the simple perturbation theory, have been derived from the following equations^[11].

$$\epsilon' - 1 = 2 J_1^2(x_{0n}) \frac{\Delta f}{f_0} R_0^2 / R_s^2 \quad (1)$$

$$\epsilon'' = J_1^2(x_{0n}) \Delta(1/Q_L) R_0^2 / R_s^2 \quad (2)$$

$$\Delta f = f_o - f_s \quad \text{and} \quad \Delta(1/Q_L) = \left(\frac{1}{Q_{Lo}} - \frac{1}{Q_{LS}} \right)$$

Where

- f_o resonant frequency;
- f_s resonant frequency with test sample;
- Q_{Lo} the loaded cavity Q-factor of empty cavity;
- Q_{LS} the loaded cavity Q-factor of cavity with test sample;
- R_o radius of cavity;
- R_s radius of sample;
- x_{on} is the nth root of $J_o(x) = 0$; and
- ϵ' and ϵ'' respectively, real and imaginary parts of the complex permittivity calculated by the simple perturbation theory.

CMS is capable of estimating, finding, and measuring the resonant frequencies f and the loaded Q-factor Q of the cavity with and without the sample. By substituting these resonance parameters, f and Q , in equations (1) and (2), CMS can calculate the two parts of the complex permittivity, ϵ' and ϵ'' and display their changes with temperature in several formats such as graphs and tables. This data in the table may be transferred to an Excel data sheet. There are two methods of the CMS calculations which are based on the length of the sample inside the cavity as shown in Fig. (4).

The term R_o^2/R_s^2 ; which represents the ratio of the cavity radius, R_o^2 , to the sample radius, R_s^2 , in equation (1) and (2), will be used by the CMS for the above calculations when the length of the sample is larger than the length of the cavity, Fig. (4-b). This ratio will be changed by the CMS or V_o/V_s when the length of the sample is shorter than the length of the cavity, Fig. (4-a). The ratio (V_o/V_s) represents the ratio of the cavity volume, V_o to the ratio of the sam-

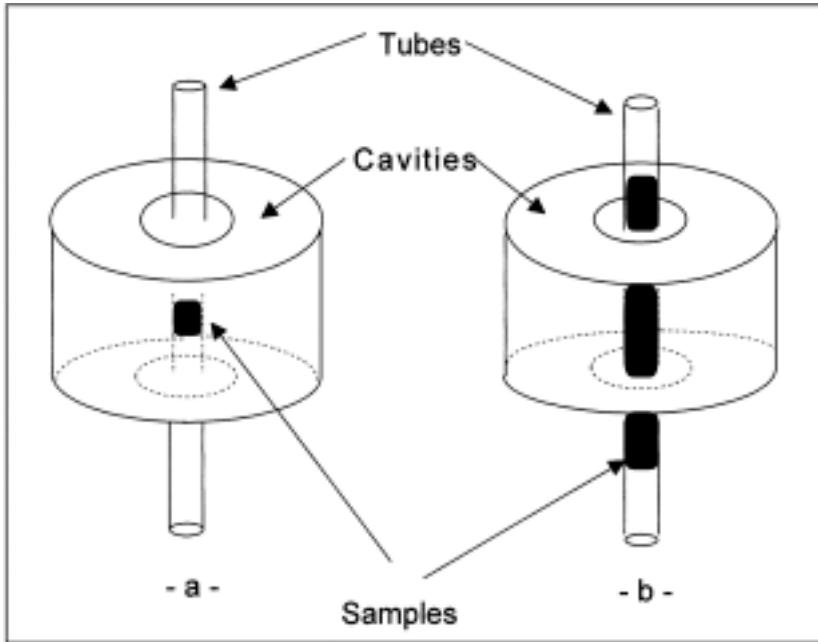


FIG. 4. Sample position in the cavity during the measurements.

ple, V_g . The CMS pathway and its functions order the system to do the measurements at high and low temperature are illustrated in Fig. (5).

Design and Construction Errors

Cavity must be cold during the measurements. There are two probabilities of rising temperature inside the cavity during the measurements at high temperature. Fig. (6) shows the system design where the cavity is positioned over the furnace and its holes are parallel to the hole of the furnace, then rising temperature from the furnace to the cavity is the first probability. The second probability is that when the sample is placed in the cavity for the measurement, the heat may come from the sample itself to the cavity. Cold water of the desired temperature is circulated in a spiral pipe which is placed between the cavity and the furnace as shown in Fig. (6). This may prevent the temperature rising from the furnace to the cavity. Preventing rising temperature from the sample into the cavity has been justified by the CMS. The CMS orders the positioner to hold and position the sample inside the cavity and at the same time it orders the VNA to estimate and catch the sample signal and record it, then orders the positioner to place the sample inside the furnace. This process takes a very short time, less than two seconds, which means that a negligible amount of heat will transfer from the sample to the cavity.

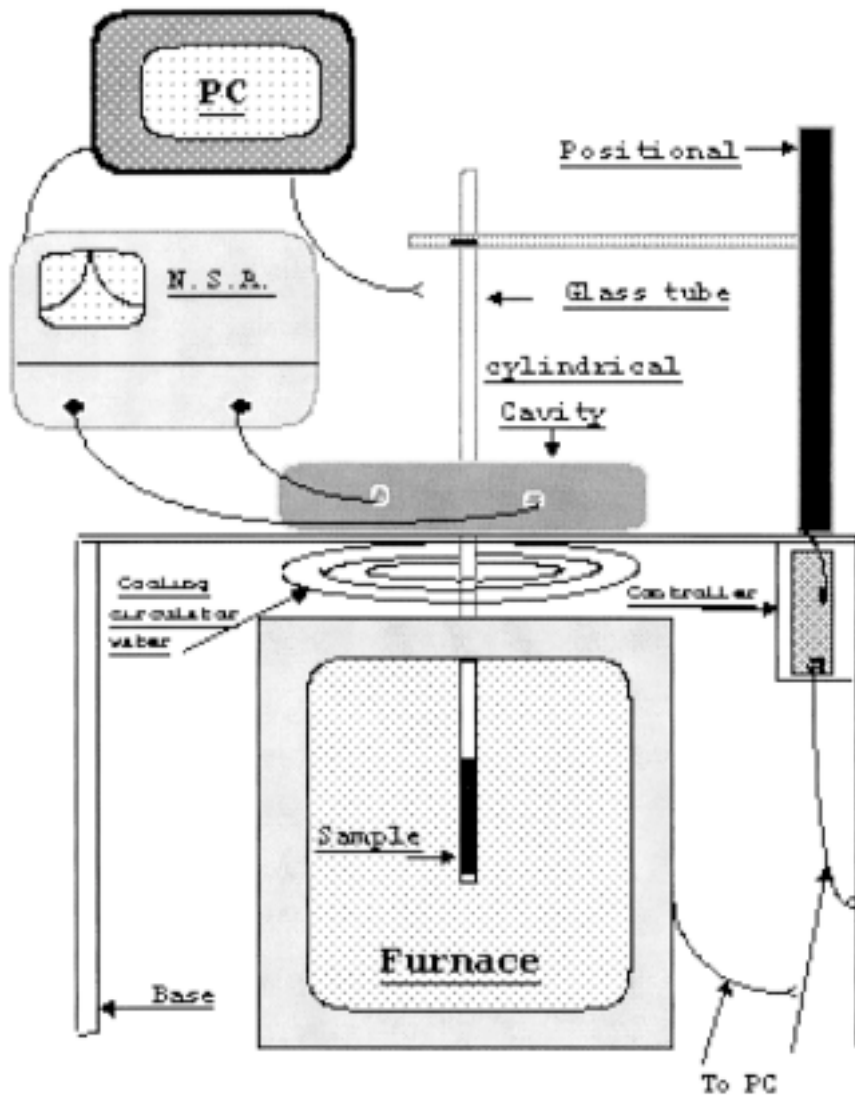


FIG. 6. The measurement system.

Measuring five modes during the desired step of temperature may cause the sample to lose heat and no longer be at the required temperature for the measurement. The CMS overcomes this problem by stabilizing the sample in the furnace, for about one minute, between each mode.

Conclusion

A microwave measurements system of the cavity perturbation technique has been designed and built at the King Abdulaziz University to contribute to the development of the microwave processing of ceramics. This system is ready to measure the dielectric properties of any kind of ceramics at five values of microwave frequencies, 615 MHz, 1412.36 MHz, 2214.15 MHz, 3017.18 MHz, and 3820.66 MHz and in the temperature range 25-2000°C in the given steps.

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جهاز قياس الخواص الكهربائية لمواد السيراميك في مدى ترددات الميكروويفز عند درجات حرارة عالية في جامعة الملك عبد العزيز قسم الطالبات

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المستخلص. لقد حصل قسم الطالبات في جامعة الملك عبد العزيز على فرصة جيدة للقيام ببحوث ما بعد البكالوريوس داخل المملكة من خلال برنامج الإشراف المشترك بين جامعة الملك عبد العزيز والجامعات البريطانية. ولقد كان هذا البرنامج ملائماً للحصول على درجة الدكتوراه دون الذهاب إلى الخارج وعلى وجه الخصوص لمحاضرات كلية العلوم، حيث يقمن المحاضرات بإنشاء معاملهم الحديثة بأنفسهم مع الإشراف من قبل المشرفين البريطانيين.

لقد أثبتت الأبحاث والتجارب العلمية أن مواد السيراميك هي من أهم المواد العازلة للحرارة ولل كهرباء مع إمكانية استخدامها داخل أجهزة الميكروويفز حيث تشغل مساحة صغيرة جداً وتعتبر مادة العصر والثورة الصناعية وأن تصنيع هذه المواد باستخدام طاقة الميكروويفز ينتج مواداً ليس لها مثيل في زمن قصير جداً فالمادة التي تصنع بواسطة الحرارة العادية في ٢٤ ساعة تصنع بواسطة الميكروويفز في ١٠ دقائق فقط. إن طاقة الميكروويفز تختلف عن جميع الطاقات الاعتيادية الأخرى فهي تعمل داخل المادة نفسها أي تخرق سطح المادة وتتحول من طاقة تحسب بالوات إلى حرارة تقدر بالدرجات المئوية داخل المادة نفسها أي إن الحرارة التي تنتج داخل المادة تعتمد على خواص المادة الكهربائية فالمادة تتعامل مع أشعة باردة أي إن أشعة الميكروويفز ليست أشعة ساخنة بل هي أشعة عند درجات الحرارة المحيطة بها. ولقد وجد إن الحاجز الذي يعيق استخدام الميكروويفز لتصنيع مواد السيراميك هو عدم القدرة في التحكم في درجات الحرارة الناتجة فهي تبدأ بداية عادية وفجأة وبدون مقدمات ترتفع

الحرارة ارتفاعا كبيرا يؤدي إلى إلحاق الضرر بالمادة وفي بعض الأحيان يؤدي إلى تدمير المادة تدميرا تاماً. إن قياس العازل الكهربائي لكل مادة على حدة عند درجات حرارة متدرجة لمعرفة درجة الحرارة التي يكون عندها العازل الكهربائي ثابتا والتي يكون عندها متغيرا يساعد في عملية التصنيع بشكل مباشر بمعنى أن التحكم في تصنيع أى مادة من مواد السيراميك بواسطة الميكروويفز لا يمكن أن يتم بدون قياس العازل الكهربائي لكل مادة على حدة. إن قياس العازل الكهربائي عند درجات الحرارة العادية متوفر وسهل جدا ولكن الصعوبة تأتي عند القياس عند درجات حرارة عالية وفي مدى ترددات الميكروويفز. وللعمل في هذا المجال كان لا بد من بناء جهاز للقيام بهذه المهمة وتأتي صعوبة بناء هذا الجهاز هو انه لا يوجد جهاز معين يستطيع قياس العازل الكهربائي عند درجات الحرارة العالية في مدى ترددات الميكروويفز وعلى من يريد أن يعمل في هذا المجال عليه تركيب الجهاز من مكونات مختلفة مكونه من عدة أجزاء مختلفة استوردت من شركات مختلفة وكان اصعب جزء بعد تجميع هذه المكونات هو ربط هذه المكونات بعضها ببعض بواسطة عمل برنامج للحاسب الآلي بحيث يجعلها تعمل أوتوماتيكاً كأنها وحدة واحدة أى أنه يأخذ القياسات عند درجات الحرارة والترددات المطلوبة ثم يحسب النتائج ويضعها في جداول ويحلل تلك النتائج بواسطة رسم العلاقات بين الثوابت والمتغيرات كل ذلك في نفس اللحظة، وكان هذا العمل جزءاً من القسم العملي للرسالة، وبفضل من الله وتوفيقه ثم بفضل مساعدة القائمين على برنامج الإشراف المشترك أصبح لدى جامعة الملك عبد العزيز ثالث جهاز في العالم لقياس العازل الكهربائي لمواد السيراميك حيث لا يوجد له مثيل سوى في كندا وجامعة نوتنجهام ببريطانيا بل إنه يتفوق عليهم من حيث الدقة والقياس عند درجات حرارة وترددات أعلى.