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Development of a microwave absorbing material based on molybdenum-doped niobium pentoxide

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Abstract

This work aimed to perform the characterization of electrical properties of niobium pentoxide, Nb_2O_5, pure and doped with molybdenum, prepared by powder metallurgy route and to do a comparative analysis of the influence of this material when sintered in a muffle or in a microwave oven. The results showed that the doping of the niobium pentoxide with 0.5% or 1% of molybdenum along with the process of sintering in a microwave oven make the material more applicable as a microwave absorber material for electromagnetic radiation in the UHF (ultra-high frequency) range between 0.3 to 3 GHz. The motivation of this work was associated with the fact that in Brazil are located two of the largest niobium reserves in the world. Under this view, the study of the processing of the ceramic materials is important considering possible technological applications.

Keywords: microwave absorbing material, niobium pentoxide, molybdenum, microwave sintering.

INTRODUCTION

More than 90% of niobium reserves of the world are in Brazil, and yet almost all the production is exported; studies on these materials and new technological applications would provide good progress for the scientific community in this field [1]. In this context, the niobium pentoxide, Nb_2O_5, is an interesting material to be investigated and has been studied for various applications; this material was chosen to be the object of this study. The microwave technology applied as sintering process of ceramic materials is a technique for material processing that has been explored and highlighted by its advantages, especially when compared with the conventional processes such as the sintering in muffle oven [2]. The electromagnetic radiation absorbing materials (RAM) is used in order to eliminate or attenuate electromagnetic radiation levels in different environments. Good progress in this area has been achieved due to the development of such materials, which have the characteristic of absorbing incident radiation and transform it into thermal energy. With the great advance in wireless communication systems, these absorptions have become increasingly important in civil and military applications [3-5]. RAM belongs to a class of materials called composite materials. In general, composites are materials composed of several phases, which exhibit properties of both constituent phases with the possibility of improvement of these properties. In engineering, it is produced by combining metals, ceramics, and polymers to produce a new generation of materials with improved characteristics [6].

The studies developed in the absorber field involve applications as the fiber impregnated in the conductive polymers matrix, fixtures for specific frequency bands and military applications in the aircraft industry [3,7-9]. In addition to the studies mentioned, different institutions developed materials for this purpose by characterizations of different chemicals and using innovative processing. The studies also include finding alternative applications for the materials that are abundant in certain regions [3, 10-12]. In the case of civil applications, the successful use of electromagnetic radiation absorbing materials is electromagnetic shielding instruments used in the manufacturing of telecommunication devices, in the electronic industry and in the medical field [13]. The main uses of these materials are related to the solution of electromagnetic interference and incompatibility problems. In this context, every electronic component that presents anomalous behavior when exposure on electromagnetic radiation justifies using RAM for protections and to have a guarantee of standard operation [14].

Several studies were carried out involving different types of composites and the addition of oxides in order to observe their effects of absorption of microwaves. It is generally carried out through the analysis of the electrical permittivity and loss tangent values. Other studies have also been made for characterizations of such properties for pure niobium and other types of ferrites for antenna applications [15-23]. To date, the system of niobium pentoxide with molybdenum has not yet been studied. This work aimed to study the viability of niobium pentoxide doped with molybdenum powder for application as an absorbing microwave compound, where, in this case, the metal particles when affected by electromagnetic waves have their molecular structure excited, and part of the incident energy is converted into heat. The influence of this doping is observed as a comparative study of the electrical properties of the pure

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niobium pentoxide. The effect of sintering by microwave and sintering in the conductive oven on its applicability as an absorbing material is also presented.

EXPERIMENTAL PROCEDURE

Samples of pure and molybdenum-doped niobium pentoxide were prepared through the powder metallurgy process, which consisted of mixing, compacting and sintering of powders. The effects of compacting pressures and sintering temperature on a muffle furnace on the electrical properties of pure niobium pentoxide have already been studied [15]. For this work, the conditions were chosen in order to obtain the highest values of permittivity and loss tangents. Doping aimed to increase the loss tangent in a particular frequency range, so the material had the characteristics of a microwave absorbing material. The microwave sintering process aimed to reduce the processing time and reducing energy costs for the sintering and coalescence of the samples. High purity powders of niobium pentoxide and molybdenum were used as can be seen in Table I.

Table I - Characteristics of niobium pentoxide and molybdenum.

Material	Manufacturer	Purity	Average particle diameter (μm)
Nb_2O_5	CBMM*	99.8%	0.4
Mo	JB Chemical	99.9%	3.5

* - *Brazilian Co. of Metallurgy and Mining*.

The concentrations used for the doping of niobium pentoxide with molybdenum were 0.5, 1 and 2%. The powder mixtures were performed in a simple spherical vessel, which was coupled to a lathe, rotating at 30 rpm for 3 h to obtain proper homogeneity. Cylindrical shape samples with dimensions of 15 mm in diameter and 7 mm in height were compacted. The powder compacting process was done by a uniaxial press at a pressure of 166 MPa. Some samples were sintered in a muffle furnace at 900 °C for 4 h and the others were sintered in a microwave oven with an operating frequency of 2.45 GHz and 900 W of power for 30 min.

After sintering, the dielectric probe method was used for the electrical characterization including permittivity and loss tangent. The Agilent vector network analyzer (85070E) with a frequency range of 0.2 to 4.5 GHz was used. Simulations were performed in a virtual environment in HFFS Ansoft software to observe the effect of inserting blocks of these materials in a waveguide as an electromagnetic radiation absorber. The waveguide was used according to the frequency band corresponding to the highest values of the complex electrical permittivity.

RESULTS AND DISCUSSION

Samples of different doping concentrations were used to find a limit of concentration for molybdenum to the niobium pentoxide. Such concentration was found when the integrity of the sintered samples was compromised in both muffle furnace and in a microwave oven. The values of permittivity and loss tangent of the molybdenum-doped niobium pentoxide were measured. The best results for application of the material as a radiation absorber were related to doping of 0.5 and 1% of molybdenum. The limit of doping for niobium pentoxide was 2% (concentration at which regions of irregularities were observed).

Measurements of electrical properties: as the complex component of electrical permittivity value is related to the losses of the material, the larger it is, the greater the material loss tangent and, hence, more energy is scattered in this frequency range. Fig. 1 shows the values of complex component of electrical permittivity as a function of operating frequency for pure and molybdenum-doped niobium pentoxide processed in muffle furnace and microwave oven. In both graphs, it is possible to observe that the samples sintered in a microwave oven presented a higher peak of the complex component of electrical permittivity, i.e. larger loss tangent peak value. These regions are the best

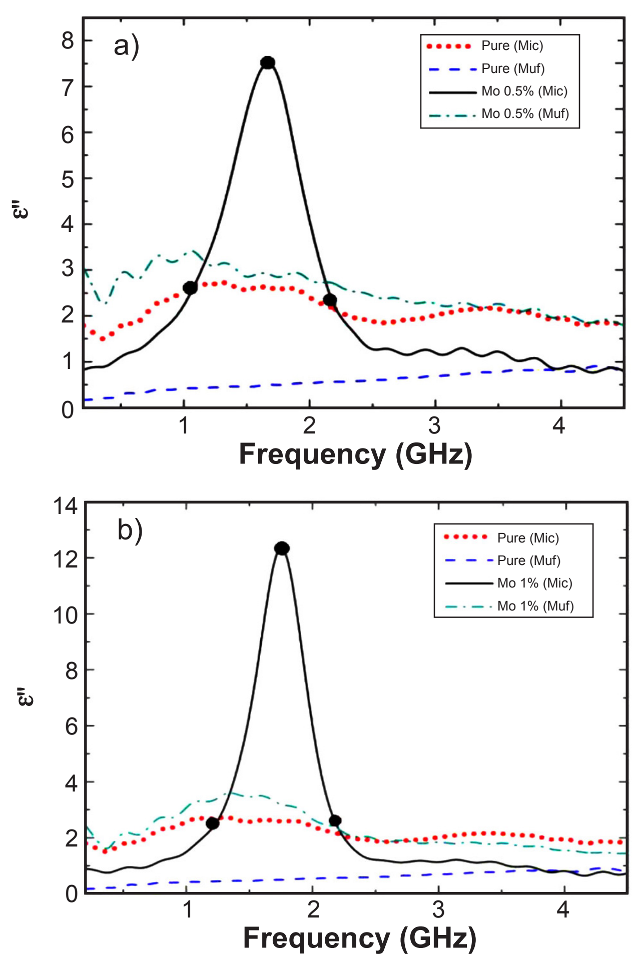

Figure 1: Values of the complex component of electrical permittivity versus operation frequency for the pure niobium pentoxide and molybdenum-doped with 0.5% (a) and 1% (b), sintered in muffle oven and microwave oven.

frequency bands for using this material for application to disperse electromagnetic energy.

Table II shows the measured values of the complex component of electrical permittivity and their respective frequencies (indicated in the graphs of Fig. 1 with dark circular marks) indicating the frequency bands of interest. According to Table II and the graphs in Fig. 1, it can be confirmed that 1% molybdenum-doped sample resulted in a higher complex component of electrical permittivity. In the case of the sintering process, sintering in the microwave oven provided the best characteristics for application as electromagnetic radiation absorbing material with higher absorption values. These values of conductivity characterize the material as a semiconductor [24]. The frequency band of interest is the UHF (ultra-high frequency), ranging from 0.3 to 3 GHz. Applications for the frequency range from about 1 to 2 GHz are broadcasting systems, radiolocation, radio navigation and the aviation industry to the amateur and mobile services [25].

Table II - Values of the complex component of electrical permittivity and their respective frequencies to the samples with 0.5% and 1% doping sintered in a microwave oven, indicated by the points in Fig. 1.

0.5% Mo		1% Mo	
Frequency (GHz)	ε”	Frequency (GHz)	ε”
1.060	2.520	1.232	2.520
1.662	7.616	1.748	12.829
2.178	2.227	2.178	2.488

Simulation: by using the higher values of the complex component of electrical permittivity and tangent loss and the best frequency bands to which it can be applied for an electromagnetic radiation absorber material, the simulation was possible with Ansoft HFFS, by placing a block of the material, processed under the best conditions to obtain the highest values of the complex component of electrical permittivity, in a waveguide for observing the efficiency of absorption. Similar studies on the electrical properties of loss tangent and electrical permittivity of materials performed simulations for analyzing the propagation of electromagnetic waves in various environments and devices, including waveguides, but none of them proposed inserting a ceramic material with metallic particles for absorption of electromagnetic radiation [26-30]. According to information provided by EIA (Electronics Ind. Assoc., USA), WR 510 waveguide which has operation bandwidth between 1.45 and 2.2 GHz was chosen. The waveguide size for this configuration is 5.1 by 2.55 inches (0.129x0.065 m). It was considered the best test body as an absorbing material. The 1% molybdenum-doped niobium pentoxide sample processed in a microwave oven was put in a point where there was a higher power dissipation. The loss tangent had a value of 1.018 and value of the complex component of electrical permittivity equal to 12.829 (the operation frequency of 1.748 GHz). For the simulation and analysis of electromagnetic wave propagation in the solid block of the material, the measured values of electrical permittivity, tangent loss, and electrical conductivity were used. To calculate the electrical conductivity, the wave propagation condition was used in environments with losses [31]. Using Eq. A, it was possible to obtain the electrical conductivity values of the analyzed samples:

$$\varepsilon'' = \frac{\sigma}{\omega}\,\varepsilon \tan\theta \qquad \text{(A)}$$

where ε” is the complex component of electrical permittivity, σ is the electrical conductivity, ε is the electrical permittivity and tanθ is the loss tangent. The values of the real and complex components of electrical permittivity, loss tangent and electrical conductivity for different samples are listed in Table III.

Simulation analysis was performed in order to observe how the radiation is absorbed by the material when it is placed as a block within the specified waveguide. Fig. 2 shows the wave propagation inside the waveguide and how it behaves when it concerns the inserted niobium pentoxide block doped with 0.5% and 1% of molybdenum. As shown, the materials act as an absorber, not allowing the passage of the wave through it. As displayed in the image, wave behavior inside the waveguide with 1% molybdenum-doped pentoxide block was similar to that with 0.5% molybdenum-doped, but in this case, as there was a lower doping, greater penetration along the block occurred, since the electrical conductivity was lower.

The sequence of images in Fig. 3a shows the wave behavior for niobium pentoxide doped with 1% of molybdenum processed in a muffle furnace. It can be seen that there is some resistance in the propagation of the wave that can be related to the doping with molybdenum, which

Table III - Values of the real and complex components of electrical permittivity, loss tangent and electrical conductivity for different samples.

	1.662 GHz frequency		1.748 GHz frequency			
	0.5% microwave	0.5% muffle	1% microwave	1% muffle	Pure microwave	Pure muffle
tanθ	0.666	0.223	1.018	0.278	0.178	0.054
ε”	7.617	2.952	12.829	3.145	2.566	0.485
ε’	11.434	13.268	12.606	11.302	14.410	8.946
σ (S/m)	0.704	0.273	1.247	0.306	0.249	0.047

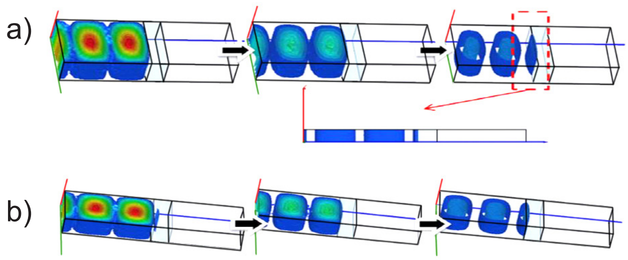

Figure 2: Sequence of images showing the propagation of electromagnetic wave in the molybdenum-doped niobium pentoxide block sintered in a microwave oven and inserted in a WR 510 waveguide, simulated in Ansoft HFFS software for 1% doping with side view detail showing the penetration (a) and 2% doping (b).

makes the passage difficult. But it can also be seen that there is also some wave passage. Such behavior may be related to the expansion of the molybdenum metal particles during the sintering in a muffle furnace, increasing the porosity of the material. According to Table III, it can be seen that the highest electric conductivity is related to the doping conditions where the sintering process was in the microwave oven, which uses a different heating mechanism for sintering; thus, there is no metal particle expansion and the porosity was not increased, which is desirable for absorbing material. In Fig. 3b another sequence of a simulated electromagnetic wave propagating images for pure niobium pentoxide sintered in a muffle furnace with the lowest electrical conductivity is shown. It is possible to see how easily the wave penetrates in the material, which was undoped and sintered in a muffle oven, and follows the propagation after this passage.

It was observed that the doping with molybdenum along

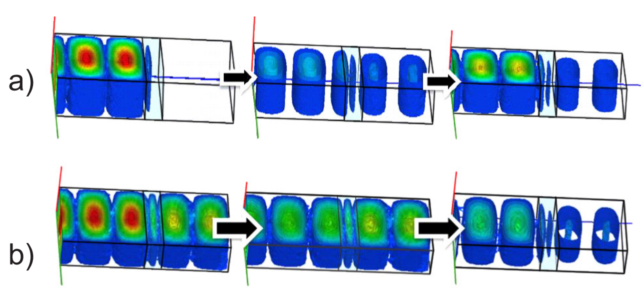

Figure 3: Electromagnetic wave propagation in 1% molybdenum-doped niobium pentoxide block (a) and pure niobium pentoxide block (b) sintered in a muffle furnace inserted in a WR 510 waveguide simulated in Ansoft HFFS software.

with sintering the material in microwave oven guarantee the obtaining a material that can be used as an electromagnetic radiation absorber. The sintering of the material in the muffle furnace increases the porosity of the material then it cannot achieve a high electrical conductivity value as obtained in the microwave sintering. The doping by itself is not sufficient to produce an electromagnetic radiation absorbing material when the sample is sintered in a muffle furnace, because there is still some passage of the waves. The microwave sintering process without doping with molybdenum does not also make the material an absorber, although it made some increases in electrical conductivity as compared with muffle furnace sintering.

CONCLUSIONS

The doping of the niobium pentoxide with molybdenum and microwave sintering processing can be used to prepare electromagnetic radiation absorbing material once they increase the electrical permittivity values, allowing more energy dissipation rate in the material. There is a certain range of frequencies where the material is most applicable as an absorber; by simulation in a selected waveguide and frequency bands where the absorption values are larger, it is possible to analyze the efficiency of a material to allow the passage of electromagnetic energy.

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