INFLUENCE OF THE SINTERING TEMPERATURE ON THE PHASES PRESENT OF YTTRIA-NIOBIA SYSTEM[1]

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Abstract
Ceramic systems are really influenced by processing parameters, especially those related with sintering. Depending of the temperature and the time input the microstructure and the final properties of the samples can be completely modified. This studied selected two compositions of the yttria-niobia system to study the influence of the temperature of sintering characterizing its phases by X ray diffraction. The parameters used were: temperatures 1200 and 1500°C for 60 hours each and samples made of 40w%Y_2O_3-60w%Nb_2O_5 and 50w%Y_2O_3-50w%Nb_2O_5. It is observed that the cubic phase increases at higher temperatures; increasing the composition of yttria, the cubic and tetragonal phases gets more evident and there are reduction on monoclinic phase for stabilization of the other ones.
Keywords: Stability phase; Y_2O_3-Nb_2O_5 system; X ray diffraction; Yttria; Niobium oxides.

INFLUÊNCIA DA TEMPERATURA DE SINTERIZAÇÃO NAS FASES PRESENTES NO SISTEMA ÍTRIA-NIÓBIA

Resumo
Sistemas cerâmicos são muito influenciados pelos parâmetros de processamento, principalmente aqueles relacionados à sinterização. Dependendo da temperatura e do tempo utilizados, a microestrutura e as propriedades finais das amostras podem ser completamente alteradas. Este estudo selecionou duas composições do sistema composto por ítria-nióbia para estudar a influência da temperatura de sinterização, caracterizando as fases presentes por difração de raios-X. Os parâmetros utilizados foram: temperaturas de 1200 e 1500°C por 60 horas e amostras processadas com as composições em peso de 40% Y_2O_3 - 60% Nb_2O_5 e 50% Y_2O_3 - 50% Nb_2O_5. Observou-se que a quantidade da fase cúbica aumenta com a elevação da temperatura; o acréscimo percentual de ítria causa aumento na presença das fases cúbica e tetragonal estabilizadas com a redução na quantidade da fase monoclínica.
Palavras-chave: Ítria; Nióbia; Temperatura de sinterização; Fases presentes.

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1 INTRODUCTION

1.1 Ceramics

The word ceramic can be traced back to the Greek term *keramos*, which means "a potter" or only "pottery". Ceramics can be defined as inorganic, nonmetallic material whose formation is due to the action of heat that activates chemical reactions; it is typically crystalline in nature and its compounds are formed between metallic and nonmetallic elements bounding.(1)
The study of ceramics consists to a large extent of methods to mitigate problems like hardness, porosity and brittleness, and accentuate the strengths of the materials, as well as to offer up unusual uses to them.
Advanced ceramics have been developed over the past half century, it's applied as thermal barrier coating to protect metal structure, wearing surfaces, or as integral components by themselves.

1.2 Sintering Process

Once powder are mixed and a ceramic bulk has been compacted, a full densification can be achieved using a process named sintering at high temperatures (up to 1.800°C), but below melting point. Its process provides energy to approximate the particles, which agglomerate by diffusion process, and diminish or even remove the porosity remains from compacting stage, shrinkage (Figure 1). The decrease in porosity is determined by the level of inicial porosity, sintering temperature and time.

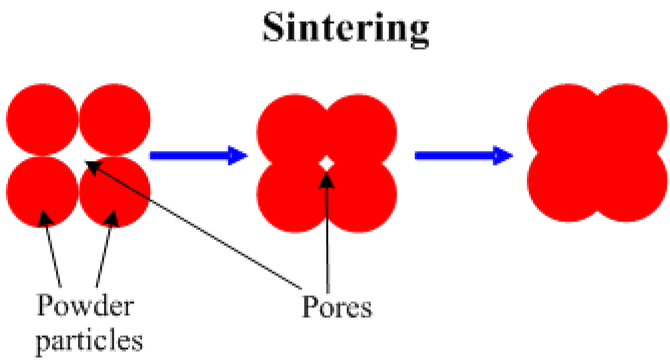

Figure 1. The sintering process, a representation.(2).

There are three stages on sintering process:(3)

- initial stage: characterized by the rearrangement of particles to increase contact points and neck formation (bonding at points with highest surface energy);
- intermediate stage: the neck grows and there is a shrinkage in the volume, the grain grows as well lengthening of grain boundaries, and the pore system becomes discontinuous;
- final stage: there is only a grain growth, the pores remaining are isolated ones and the grain boundary pores eliminated.

The driving force of sintering process is the reduction of surface energy of the particles causing by the decreasing of vapor-solid interfaces.

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All the characteristics associated to temperature like phase transformation occurs during sintering. The result of densification is an improvement of mechanical properties.

1.3 Y_2O_3 – Nb_2O_5 Phase Diagram

The phase diagram of Y_2O_3-Nb_2O_5 system has been poorly understood. The diagram used is from 1989 (Figure 1),[4] and it was built after chemical and gravimetric analyses using X-ray diffraction and DTA. The solubility limits of Nb_2O_5 in Y_2O_3 and of Y_2O_3 in $YNbO_4$ have not been determined properly.

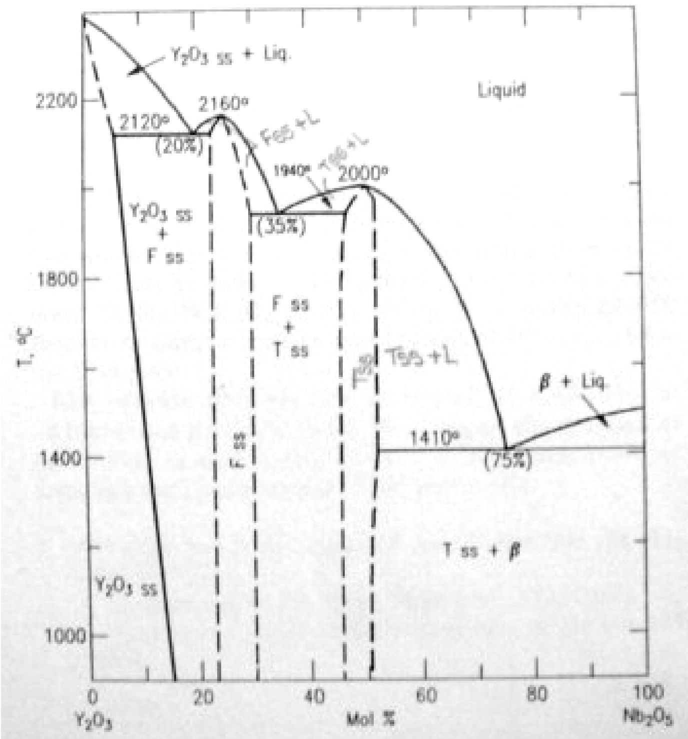

Figure 1. Phase diagram of Y_2O_3-Nb_2O_5 system accepted on literature.[4]

The purpose of this work is to see if the sintering temperature influences on the phase transformation of the system made of Y_2O_3 and Nb_2O_5, and how does it affect the properties of the material.

2 EXPERIMENTAL PROCEDURE

We used powders of Y_2O_3 and Nb_2O_5 with high level of purity. The compositions used are listed on Table 1 and showed on Figure 2.

Table 1. Compositions used to prepare the samples

Compositions (mol %)	
Nb_2O_5	Y_2O_3
50	50
60	40

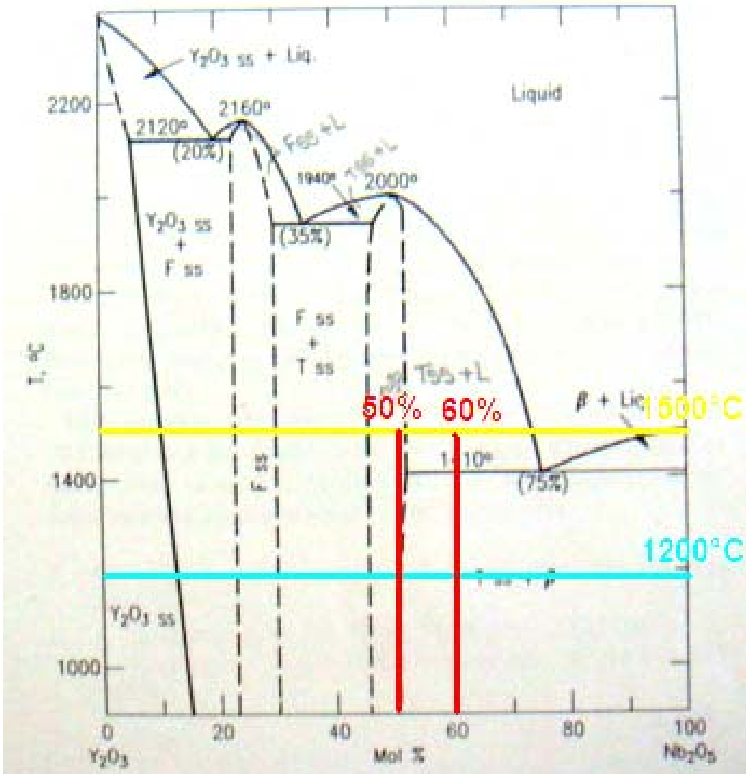

Figure 2. Compositions used on the experiment and identified on the phase diagram.

It was used a chemical route to prepare the samples, it consist on mixing the oxides in a ball mill, homogenization on water and sinter at high temperatures. It was used two different sinter temperatures: 1.200°C and 1.500°C, the samples were left for sixty (60) hours on the mufla oven to the correspondent temperature.

The bulk was milled and the power obtained was taken to X ray analyses. The diffraction pattern was analyzed using Powder Cell software and the phases identified.

To identify the phases presented it was used the information of patterns obtained from PDFWin database and also from handbook.

3 RESULTS

The Figures 3 and 4 present the comparison between the difractograms obtained for both analyses. On them there are specified the phases of each peak.

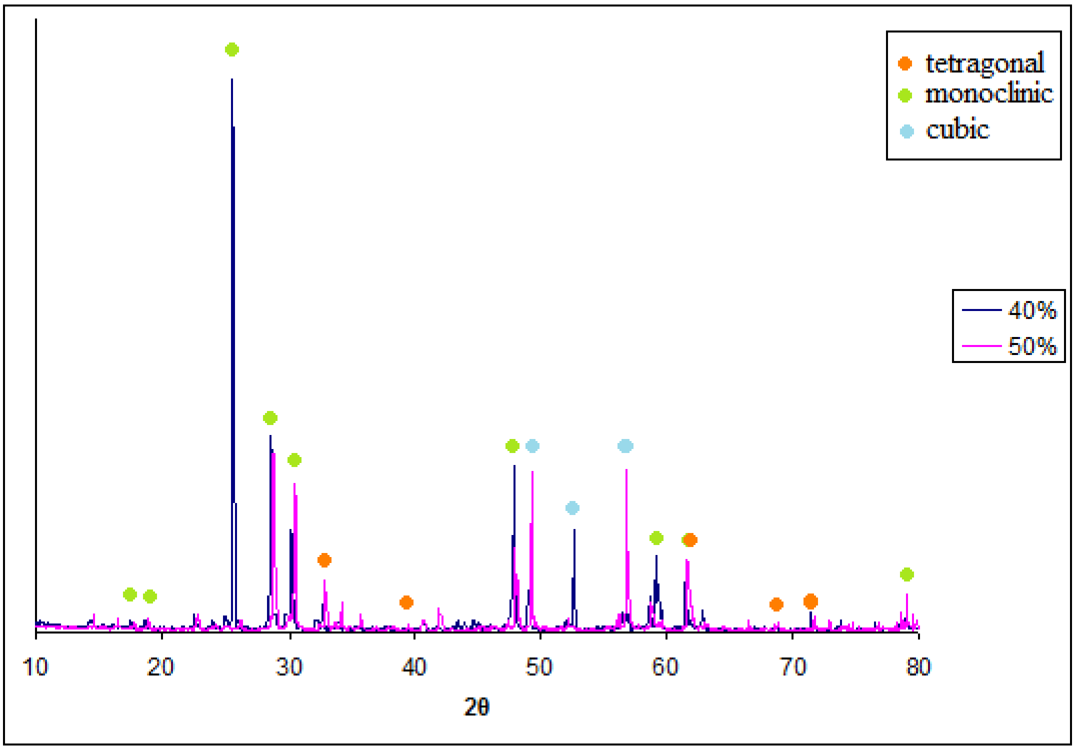

Figure 3. Comparative difractograms between 40% e 50% em mol de Y_2O_3 processed at 1500°C for 60 hours.

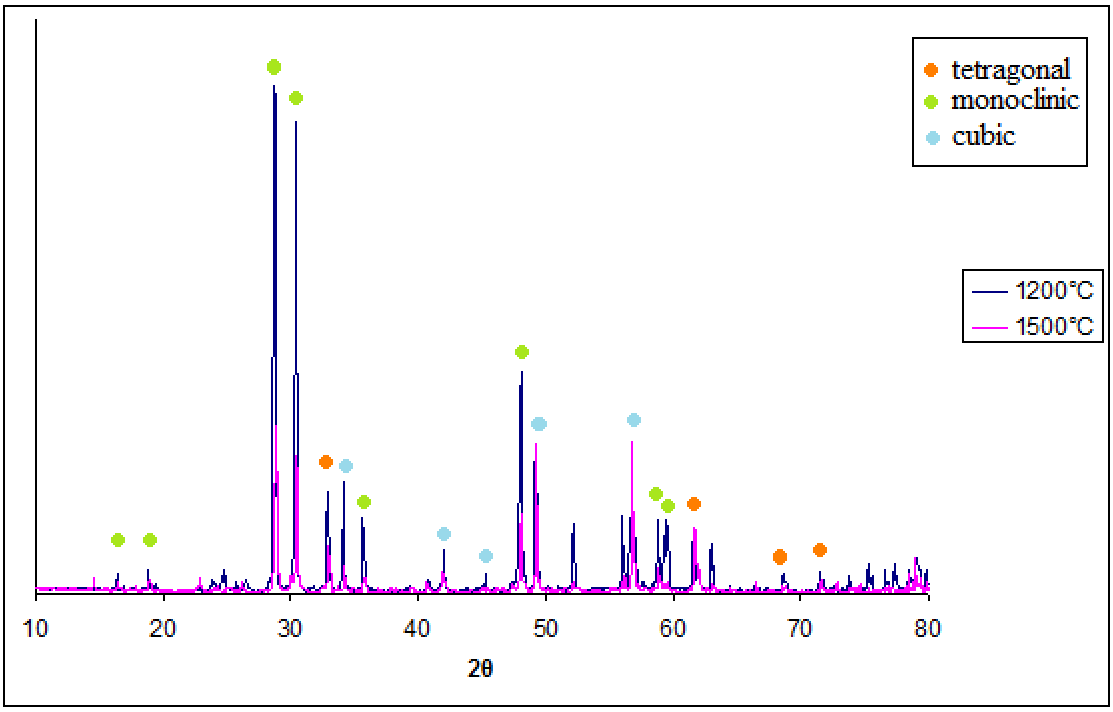

Figure 4. Superposition of the difractograms obtained for different sinter temperature (1200° and 1500°C) for 50% mol de Nb_2O_5 processed at 1500°C for 60 hours.

4 DISCUSSION

The effect on temperature and composition can determine how the material can behave. It's possible to see in the Figures 3 that the reducing of 10% mol of niobia, the monoclinic phase, characteristic of this oxide, reduces its amount, even

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disappearing from some angles (like the one present around 27° at 40%, but omissible for 50%). There is also an increase on the intensity of cubic and tetragonal phases. According to the phase diagram on figure 2, there wasn't expected to have cubic phase at these compositions, but the software analyses results the phase with higher probably to correspondence at the angles, however it doesn't exclude the possibility of these points correspond to the tetragonal phase, that has coincident angles with the cubic one.
On figure 4, the higher temperature applied to the system changes mainly the cubic and the tetragonal phases, remaining the monoclinic one with lower intensity. It happens because the presence of yttria changes the microstructure of the bulk, before dominated by monoclinic niobia solid solution.

5 CONCLUSION

It is concluded that the amount of oxides and the temperature are the main factors to achieve a certain crystalline structure on ceramics. The temperature has higher effect than composition, and only the increase of 300°C can change completely the quantitative phases presented. The system has monoclinic phase domain for higher amounts of niobia, and at equal amount of oxides, the higher temperature applied, less intensity has the monoclinic phase, but higher is the presence of cubic and tetragonal phase.

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REFERÊNCIAS

1 Available in: http://www.uio.no/studier/emner/matnat/kjemi/KJM3100/v06/undervisningsmateriale/kjm3100_ceramics_f.pdf
2 Available in: http://www.substech.com/dokuwiki/doku.php?id=sintering_of_ceramics
3 Available in: http://www.eng.um.edu.mt/dmme/documents/4001s8.pdf
4 BABICH, T.G.; ZAGORODNYUK, A.V.; TETERIN, G.A.; SADKOVSKAYA, L.V.; ZHIRNOVA, A.P. Sov. Progr. Chem., 55 [2] 21-24, 1989.