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Production Of Nbc from Nb_2O_5 in a rotating cylinder reactor: Kinetic study of reduction/carburization reactions

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ABSTRACT

The present work aims at a kinetic model for converting NbO_2 into NbC in the isothermal region of the second reduction/carburization reaction. The model presented for the solid phase was based on the variation of the mass obtained for experiments with reactions interrupted at different times and processed at temperatures of 1148, 1173 and 1223 K. The reaction took place in a rotating cylinder reactor, with rotation velocity of 5 rpm, gas phase flow of $4.033 \times 10^6\ m^3/s$ and initial mass of Nb_2O_5 0.004 kg (nominal).

The conversion of NbO_2 was found from the mass balances performed based on the set of stoichiometric equations of the reduction/carburization of Nb_2O_5 into NbC and used to validate the nucleation model using two parameters (N, M) which indicate, respectively, the tridimensional growth of the nuclei formed and a fourfold increase in the growth velocity of the nuclei.

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1. Introduction

Thermodynamics predicts the synthesis of niobium carbide through solid–solid reactions ($Nb_2O_5 + 7C \rightarrow 2NbC + 5C$) to happen under the following conditions: free energy $\Delta G = 0$ and pressure $P = 0.1$ MPa at a minimum temperature of $T = 1228$ K and enthalpy $\Delta H = +4928$ kJ/kg. Although thermodynamics cannot predict when a reaction will occur, it can predict whether the reaction is truly feasible in practice. Activation energy, diffusion resistances and kinetic considerations about the reaction are the elements that allow the occurrence of a reaction to be predicted. The reduction and carburization of metallic oxides in high temperature rotating ovens have been reported in patents, however applications are still in the development stage. Descriptions of rotating ovens have been cited only in small-scale operations or have focused on theoretical aspects such as heat transfer or material transportation. It is expected that, once perfected, high temperature rotating ovens will produce excellent, consistent, reproducible, and economic carbide powders, which are superior to those currently on the market. In practice, the production of commercial NbC in a fixed bed reactor using a solid–solid reaction occurs at temperatures around 2000 K, Weimer [1].

Different nanostructured carbide synthesis pathways for applications in advanced technologies have been proposed in recent years based on the conversion of various precursors, by means of gas–solid reactions using a flow mixture of CH_4/H_2. However, few studies have investigated the kinetics of these transformations. Niobium carbide synthesis through a fixed bed gas–solid reaction from the Nb_2O_5 precursor was partially investigated by Teixeira da Silva et al. [2]. The kinetics of the first reduction reaction from Nb_2O_5 to NbO_2 was developed mainly in the temperature ramp zone and agreed with the nucleation model.

The present study shows the results of a kinetic model in converting Nb_2O_5 to NbC in the isothermal region of the second reduction and the carburization reaction of NbO_2 to NbC. This first part of the model approached the solid phase, obtaining parameters (M) and (N) of Avrami's nucleation model, Avrami [3,4]. Other model described in the literature (unreacted core model, Levenspiel [5]) was tested for the solid phase but the results did not agree with the ones of this work.

2. Materials and methods

The following gases were used: CH_4 (99.99%), special argon (AGA) and synthetic air (White Martins). All were passed through purifiers containing phosphorous pentoxide to remove humidity. The niobium pentoxide Nb_2O_5 (99.9%) and the niobium carbide NbC (99.46%) were used as supplied by Alfa Aesar (Johnson Matthey Company).

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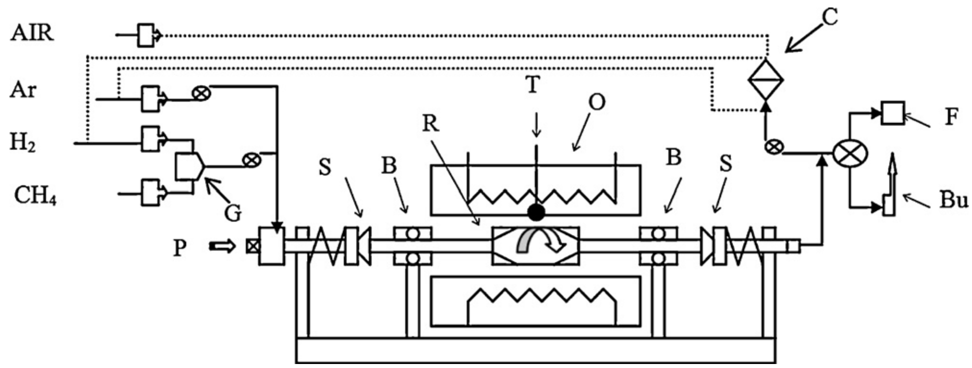

Fig. 1. Experimental device.

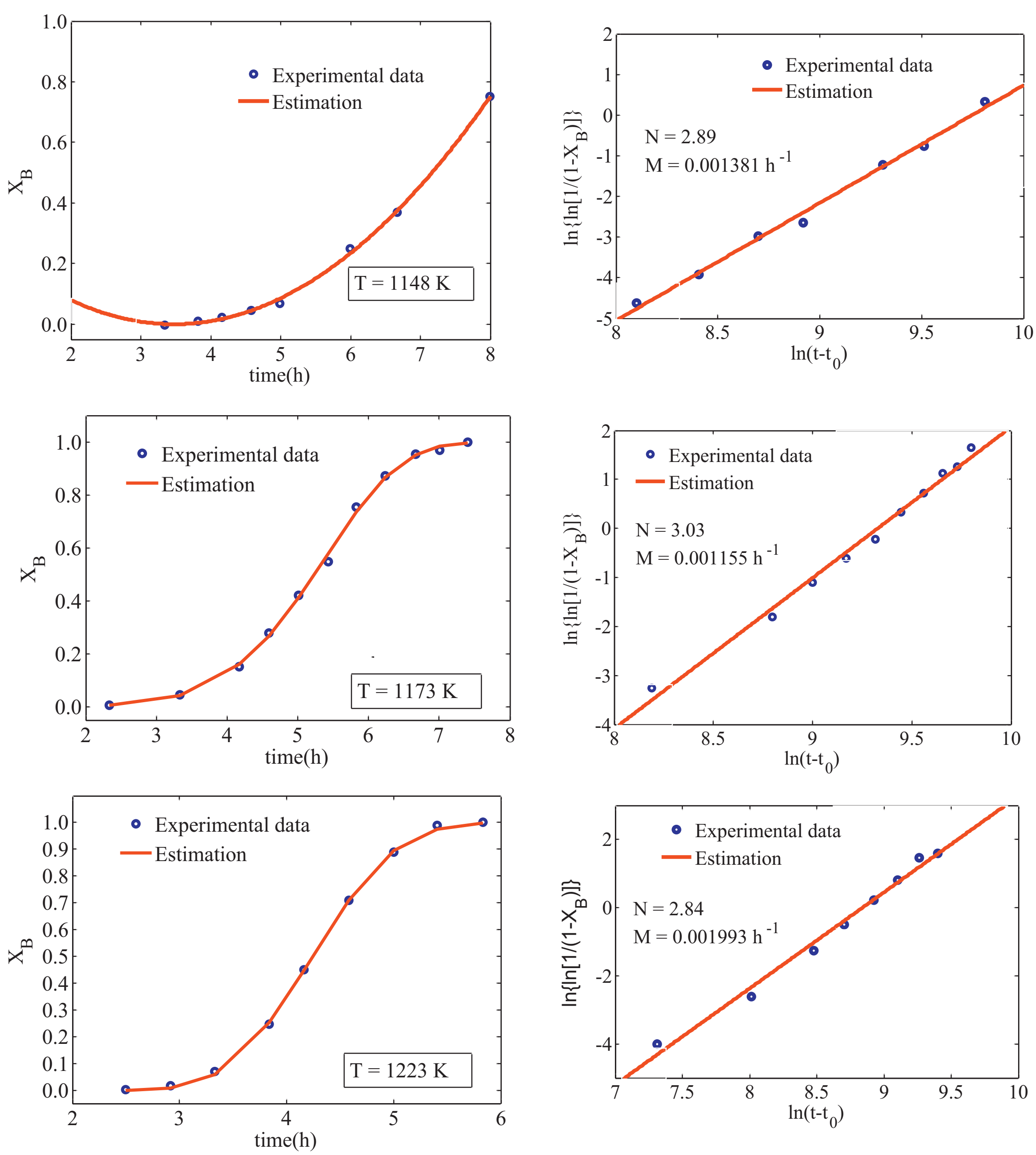

Fig. 2. Conversion of NbO_2 to NbC at different temperatures: determination of Kinetic Parameters of Avrami's Model.

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