Pergamon

Materials Research Bulletin, Vol. 31, No. 1, pp. 55-61, 1996

0025-5408/96 $15.00 + .00

0025-5408(95)00160-3

PREPARATION OF NICKEL OXIDE POWDER BY DECOMPOSITION OF BASIC NICKEL CARBONATE IN MICROWAVE FIELD WITH NICKEL OXIDE SEED AS A MICROWAVE ABSORBING ADDITIVE

Yan Wang and Jia-Jun Ke
Institute of Chemical Metallurgy, Chinese Academy of Sciences,
P. O. Box 353, Beijing 100080, P. R. China

(Received June 21, 1995; Refereed)

ABSTRACT

Nickel oxide(NiO) powder is prepared by decomposition of basic nickel carbonate ($mNi(OH)_2.nNiCO_3.xH_2O$) in microwave field with NiO seed as a microwave absorbing additive. Basic nickel carbonate(BNC) can decompose completely to NiO powder in a short time. Firstly, the heat for BNC decomposition is provided by NiO seed which absorbs microwave and then by NiO product which also absorbs microwave. The decomposition process of BNC can be accelerated by increasing the amount of BNC, the amount of NiO seed or the microwave field power. The size of NiO powder product is about 180nm when the size of BNC used is about 160nm.

MATERIAL INDEX: basic nickel carbonate, nickel oxide.

INTRODUCTION

Microwave heating(1,2), a dielectric heating process, has attracted a lot of attention and is widely studied in the sintering of advanced materials, chemical analysis and chemical synthesis for its distinctive characteristics, e.g., cost saving, selective heating and less contamination to sample. The rate of temperature rising of a material due to the electric field of microwave irradiation is determined by the characteristics of the material, such as the density, the specific heat capacity and the dielectric loss, as well as the the intensity and frequency of electric field. Therefore, microwave heating is a selective heating process. The material which has high dielectric loss can be heated by microwave irradiation, and it is called microwave absorbing material or microwave susceptible material. On the other hand, the material which has low dielectric loss cannot be heated by direct microwave irradiation, and it is called microwave poorly absorbing material or microwave transparent material. In the

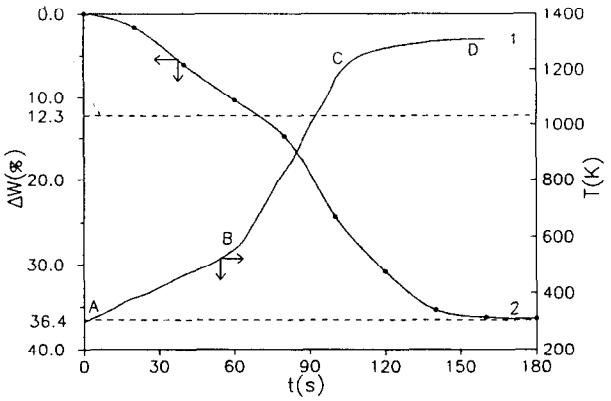

FIG. 1.

Heating process of the mixture sample and the decomposition process of BNC (P = 700W, W_{s0} = 2.00g, A = 0.4). 1—temperature of the mixture sample; 2—weight loss of BNC.

later case, indirect heating appears to be required(3) . Indirect heating involves the use of a microwave absorbing material, which is in turn used to heat a poorly absorbing sample. One method is placing the sample in a powder bed of high dielectric loss powder, such as Al_2O_3+Fe_3O_4(4) and silicon carbide (SiC)(5,6). If the product of a material treating in microwave field has high dielectric loss, it can be used as a microwave absorbing material directly. For example, a fluidized bed consisting of UO_2,U_3O_8 and UO_3 powder is used for treating uranyl nitrate solution to obtain UO_2 by microwave heating(7).

Nickel oxide is widely used in the production of catalyst and other nickel salts. The preparation of nickel oxide was widely studied. Thermal decomposition of basic nickel

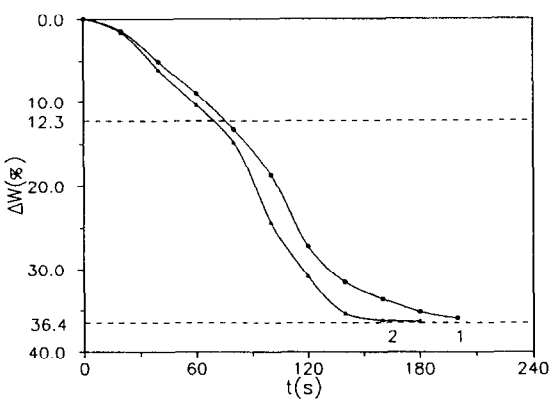

FIG. 2.

Effect of the amount of mixture sample on the decomposition process of BNC (P = 700W, A = 0.4) $W_{s0}(1+A)$(g): 1 - 1.40; 2 - 2.80.

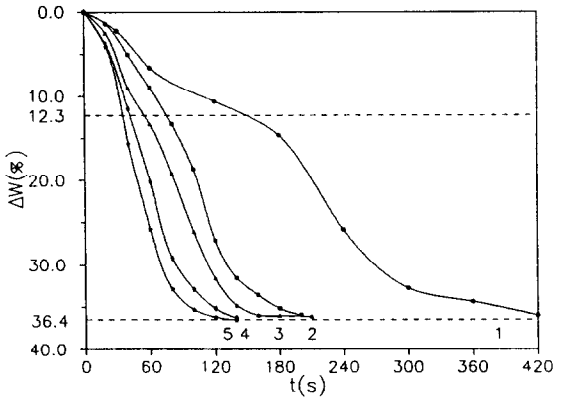

FIG. 3.
Effect of the amount of NiO seed on the decomposition process of BNC (P = 700W, W_{s0} = 2.00g) Ratio A: 1 - 0.2; 2 - 0.4; 3 - 0.6; 4 - 0.8; 5 - 1.0.

carbonate ($mNiCO_3.nNi(OH)_2.xH_2O$) was studied using thermogravimetry(TG) by some researchers(8,9). They concluded that the thermal decomposition proceeded in two steps, i.e., the evolution of crystal water occurred in the first step and the evolution of H_2O and CO_2 from the anhydride ($mNiCO_3.nNi(OH)_2$) occurred in the second step.

The authors' prior work shows that BNC irradiated by microwave directly can remove most of its crystal water, but can not decompose to NiO completely. If SiC is used as an indirect heating medium, BNC can decompose to NiO completely(6).

Nickel oxide(NiO) is a microwave absorbing material, and can be heated quickly in microwave field(6,10). The present work is to add NiO seed (it is called "seed" here because

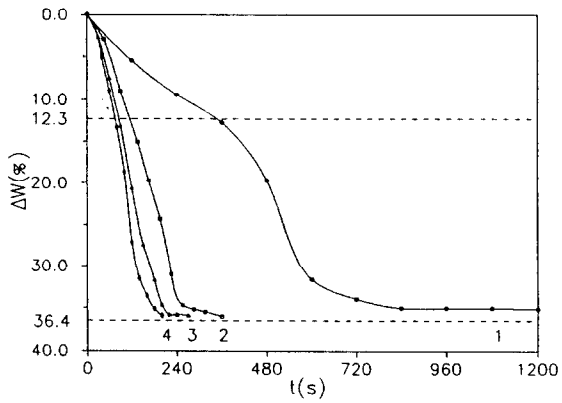

FIG. 4.
Effect of the microwave field power on the decomposition process of BNC (W_{s0} = 2.00g, A = 0.4) P(W): 1 - 220; 2 - 450; 3 - 580; 4 - 700.

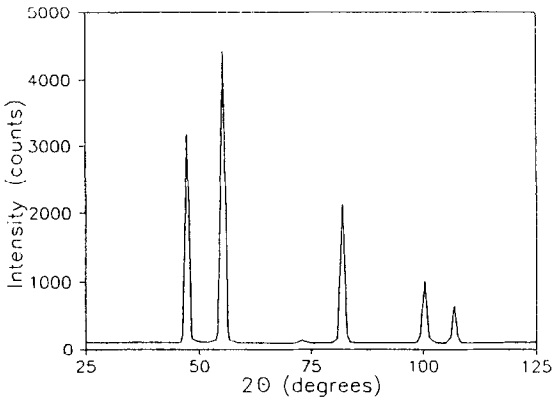

FIG. 5.
XRD patterns of the product of BNC decomposition.

it initiates the process) (11) directly to BNC, then the mixture is treated in microwave field. BNC can decompose completely to NiO in a short time.

EXPERIMENTAL

Samples. Analytical reagent grade BNC is used. Its $NiO:CO_2:H_2O$ molar ratio (1.00:0.39:1.40) is determined by chemical analysis, and it is almost equal to the ideal composition $2NiCO_3.3Ni(OH)_2.4H_2O$(1.00:0.40:1.40). The size of BNC is determined as 160nm by scanning electron microscopy(SEM). NiO seed is prepared by calcination of BNC in a muffle furnace under 873K for 3h, and its size is about 180nm.

A series of mixtures with different amount of NiO seed and BNC are prepared as the sample. The amount of NiO seed is indicated with the ratio A of the amount of NiO seed W_{NiO} to the amount of BNC W_{BNC}($A=W_{NiO}/W_{BNC}$).

Microwave Heating. A microwave oven of 700W and 2.45GHz(Type 5250, National Co.,Japan) is used in the experiments. The power of the microwave oven P is adjusted by generating continuous or pulse mode microwave with different pulse time. Modification is made to the microwave oven to insert thermocouple for temperature measurement. A type K thermocouple sheathed in stainless steel and a recorder(Type U--125MU, Shimadzu Co., Japan) are used for continuous temperature measurement.

The mixture is weighed as $W_{S0}(1+A)$ to keep the amount of BNC for each experiment W_{S0} the same. Then the mixture is placed in a 12ml quartz crucible which is placed in a certain position in the microwave oven. A stop-watch is used for timing. After treating a certain time, the crucible containing the product is put into a dryer immediately. The product is weighed after cooling.

The decomposition degree of BNC is indicated with its weight loss $\Delta W(\%)$:

$$\Delta W = \frac{W_{S0}(1 + A) - W_S}{W_{S0}} \times 100\%$$

where W_{S0} is the weight of BNC in the mixture sample before treatment, $W_{S0}(1+A)$ is the weight of mixture sample before treatment, and WS is the weight of product after treating for a certain time.

RESULTS AND DISCUSSION

Heating Process of the Mixture Sample and Decomposition Process of BNC. The mixture of NiO and BNC is treated in the microwave field. The experimental results are shown in FIG.1.

It can be seen from FIG.1 that with the microwave irradiation the temperature of mixture increases quickly (A--B), then increases more quickly(B--C), and at last increases slowly(C--D). The curve describing the weight loss vs. time doesn't show the two steps which were observed in the prior experiments with SiC as an indirect heating medium(6). At the beginning of treating process, the rising temperature of mixture is due to the NiO seed absorbing microwave. With the decomposition of BNC, NiO is produced, then the rising temperature of sample is due to the NiO product as well as the NiO seed absorbing microwave during this period, the temperature increases more quickly. At last, the temperature remains at about 1350K and the decomposition process of BNC is completed. Because the temperature

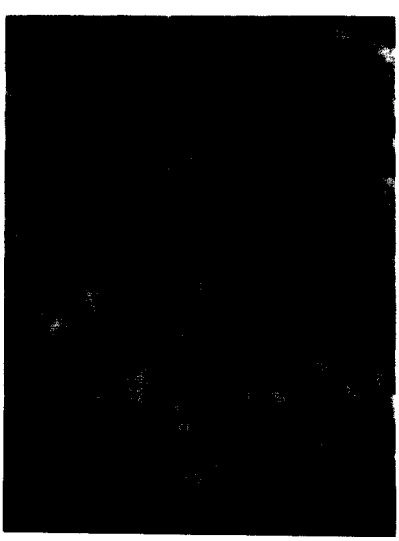

FiG. 6.
Typical SEM micrograph of the NiO product.

increases quickly, the step of BNC losing crystal water and the step of the anhydride ($2NiCO_3.3Ni(OH)_2$) losing H_2O and CO_2 overlap.

Effect of the Amount of Mixture Sample on the Decomposition Process of BNC. Experiments are carried out to investigate the effect of the amount of mixture sample on the decomposition process of BNC. The experimental results are shown in FIG.2.

It can be seen from FIG.2 that higher amount of mixture sample causes higher decomposition rate of BNC. Because the amount of sample is small, it is supposed that the change of electric intensity caused by the change of sample amount is small. For a sample, the heat produced by absorbing microwave energy per a unit volume sample is consistent and has no relationship to the amount of sample, but the heat lost to the environment per a unit volume sample is smaller if the sample has larger volume. Therefore, the sample with higher amount and larger volume has higher temperature in a same microwave treating time, and the decomposition rate of BNC is faster.

Effect of the Amount of NiO Seed on the Decomposition Process of BNC. Experiments are carried out with mixture sample containing different amounts of NiO seed and certain amount of BNC to investigate the effect of the amount of NiO seed on the decomposition process of BNC. The experimental results are shown in FIG.3. It can be seen from FIG.3 that higher amount of NiO seed accelerates the decomposition process of BNC. This is due to the high dielectric loss of NiO itself.

Effect of the Microwave Field Power on the Decomposition Process of BNC. The effect of microwave field power on the decomposition process of BNC is shown in FIG.4. It can be seen from FIG.4 that higher microwave field power causes higher decomposition rate of BNC.

The decomposition product of BNC is identified as NiO with chemical analysis(Ni content is 78.57%) and X--ray diffraction(XRD) analysis (FIG.5). The size of NiO powder product is about 180nm determined by SEM(FIG.6).

CONCLUSIONS

Nickel oxide(NiO) powder can be prepared by decomposition of basic nickel carbonate ($mNi(OH)_2.nNiCO_3.xH_2O$) in microwave field with NiO seed as a microwave absorbing additive. Basic nickel carbonate(BNC) can decompose completely to NiO powder in a short time. Increasing the amount of BNC, the amount of NiO seed or the microwave field power can accelerate the decomposition rate of BNC. The size of NiO powder product is about 180nm when the size of BNC used is about 160nm.

ACKNOWLEDGEMENTS

The authors thank the National Nature Science Foundation of China and Beijing Zhongguancun Associated Center of Analysis and Measurement for the financial support.

REFERENCES

1. R. Roy, S. Komarneni and L. J. Yang, J. Am. Ceram. Soc., 68, 392(1985).
2. F. Selmi, S. Komarneni, V. K. Varadan and V. V. Varadan, Mat. Lett., 10, 235(1990).

3. H. Sutton, Microwave processing of Materials III (Mat. Res. Soc. Symp. Proc. Vol.269), p.3. Material Research Society, Pittsburgh, Pennsylvania(1992).
4. G. Bram, A. Loupy, M. Majdoub and A. Petit, Chem. \& Ind., (6),396(1991).
5. I. Ahmad, R. Dalton and D. Clark, J. Microwave Power and Electromag. Energy, 26(3), 128(1991).
6. Y. Wang and J. J. Ke, Proceedings of the 2nd International Conference ``Non-Ferrous Metals' 95", September 7--9, 1995 in Krakow, Poland.
7. Tokyo Shibaura Denki KK, Jpn. Patent JP58194742(1983).
8. R. M. Mallya and A. R. V. Murthy, J. Indian Inst. Sci., 43(3), 131(1961).
9. N. A. Cathey, D. H. Kerridge and S. A. Tariq, Thermochim. Acta, 61, 185(1983).
10. D. M. P. Mingos and D. R. Baghurst, Chem. Soc. Rev., 20, 1(1991).
11. S. Komarneni and R. Roy, Mat. Lett., 4, 107(1986).