Synthesizing higher nitride of molybdenum (Mo) and iron (Fe) in ammonia (NH_3) gas stream under irradiation of concentrated solar beam in a solar furnace

Erzeugung von hoch nitridhaltigen Molybdän- und Eisennitriden in einem Ammoniumgasvolumenstrom bei Bestrahlung durch konzentrierte Solarstrahlen in einer solaren Brennkammer

N. Shohoji[1], F. Almeida Costa Oliveira[1], J. Cruz Fernandes[2], L. Guerra Rosa[2], J. Rodríguez Garcia[3], I. Canadas Martínez[3], C. Ramos Miñarro[3], T. Magalhães[1], F. Cestari[4]

Flowing gaseous ammonia NH_3 with suppressed extent of dissociation (un-cracked NH_3) is acknowledged to function as a powerful nitriding medium to realize formation of metal nitride MN_x with considerably high N/M ratio x that cannot be achieved through reaction of M with N_2 gas. For example, mono-nitride δ-MoN of Mo and ε-FeN_x phase of Fe with x = 0.33 ~ 0.50 (i.e. hypo-stoichiometric sub-nitride ε-Fe_2N) were reported to be difficult to prepare in N_2 gas environment even at elevated pressure but might be synthesized in flowing NH_3 gas at normal pressure when reaction temperature and NH_3 gas flow rate were set adequately. In the present work, nitriding experiments for Mo and Fe were carried out in flowing NH_3 gas under irradiation with concentrated solar beam. The acquired experimental evidences demonstrated that temperature range for formation of δ-MoN was somewhat extended in flowing NH_3 gas under heating with concentrated solar beam compared with that under heating in conventional laboratory or industrial electric furnace. On the other hand, no such merit of extending temperature range for formation of ε-Fe_2N in flowing NH_3 gas was detected in the present work under heating with concentrated solar beam.

Keywords: solar furnace / flowing gaseous ammonia (NH_3) / nitride / molybdenum (Mo) / iron (Fe)

Schlüsselwörter: Solarbrennkammer / Ammoniumgasvolumenstrom (NH_3) / Nitride / Molybdän (Mo) / Eisen (Fe)

1 Introduction

Two pioneering works using flowing ammonia (NH_3) gas as a nitriding medium were published almost simultaneously in a year 1930 in Germany; one was by Hägg demonstrating possibility of synthesizing mono-nitride of molybdenum, δ-MoN, that could not be prepared through reaction of molybdenum (Mo) with nitrogen (N_2) gas and another by Lehrer reporting systematically the extent of nitriding in the synthesized FeN_x phases as functions of temperature T and extent α(NH_3) of dissociation of flowing gaseous ammonia NH_3 [1, 2]. For nitride formation from iron (Fe) in ammonia (NH_3) gas flow, Jack reported further details later by characterizing the synthesized phases using X-ray diffraction (XRD) while phase identification in the reaction product in the Lehrer's work was done by magnetic measurement [2, 3]. In the work by Hägg, phase identification for reaction products from molybdenum (Mo) was made by X-ray diffraction using CrKα radiation [1].

Regarding δ-MoN synthesis under flow of ammonia (NH_3) gas, Katsura and collaborators demonstrated later that δ-MoN formation was very sensitive to reaction temperature T showing that δ-MoN phase started to emerge in the reaction product being co-existed with sub-nitride Mo_2N under reaction at T = 700 °C [4]. The formed δ-MoN dissociated away when the reaction temperature T was risen by mere 25 °C to T = 725 °C. The δ-MoN formation behavior did not seem to be very critically affected by ammonia (NH_3) gas flow rate in the examined range between 50 ml/min (corresponding to linear velocity ~7 cm/min in the horizontal tubular furnace used in the experiment) and 500 ml/min (lin-

[1] Laboratório Nacional de Energia e Geologia, Laboratório de Energia, Estrada do Paço do Lumiar, 22, 1649-038 Lisboa, Portugal
[2] Universidade Técnica de Lisboa, Instituto Superior Técnico, Departamento de Engenharia Mecânica, Av. Rovisco Pais, 1049-001 Lisboa, Portugal
[3] Plataforma Solar de Almería, Centro de Investigaciones Energética, Medioambientales y Tecnológicas, Apartado 22, E-04200 Tabernas (Almería), Spain
[4] Università degli Studi di Trento, Dipartimento di Ingegneria dei Materiali e Tecnologie Industriali, via Mesiano 77, 38123, Trento, Italy

Corresponding author: N. Shohoji, Laboratório Nacional de Energia e Geologia, Laboratório de Energia, Estrada do Paço do Lumiar, 22, 1649-038 Lisboa, Portugal
E-mail: nobumitsu.shohoji@lneg.pt

