International Journal of Chemical, Materials and Biomolecular Sciences
ISSN: 2415-6620
Vol:11, No:7, 2017

Synthesis of Mg/B Containing Compound in a Modified Microwave Oven

Gülşah Çelik Gül, Figen Kurtuluş

***Abstract*—**Magnesium containing boron compounds with hexagonal structure have been drawn much attention due to their superconductive nature. The main target of this work is new modified microwave oven by on our own has an ability about passing through a gas in the oven medium for attainment of oxygen-free compounds such as c-BN. Mg containing boride was synthesized by modified-microwave method under nitrogen atmosphere using amorphous boron and magnesium source in appropriate molar ratio. Microwave oven with oxygen free environment has been modified to aimed to obtain magnesium boride without oxygen. Characterizations were done by powder X-ray diffraction (XRD), and Fourier transform infrared (FTIR) spectroscopy. Mg containing boride, generally named magnesium boride, with amorphous character without oxygen is obtained via designed microwave oven system.

***Keywords*—**Magnesium containing boron compounds, modified microwave synthesis, powder X-ray diffraction, FTIR.

I. Introduction

MAGNESIUM boride is a type of boride compound containing Mg and B elements. MgB_x (x = 2, 4) obtained in 1953 with hexagonal structure for the first time. Although the MgB_2 compound is an older compound [1], it has not been discovered until 2001 that it exhibits superconductivity properties. It was discovered in 2001 in an international conference in Japan by Akimitsu et al., that MgB_2 shows superconductivity at 39 ° K [2]. Magnesium boron is interesting compound which have a hexagonal structure because of its inexpensive cost, high T_c critical temperature, simple crystal structure, large coherence length, high critical current and area density, lower anisotropic property, and current flow facilitating grain boundaries [3]. The transition to superconductivity at 39 °K of MgB_2, an intermetallic compound, has made this material a new alternative for existing superconductivity applications [4].

The MgB_2 compound is made between the hexagonal layers of the successive magnesium atoms and hexagonal plane layers of boron atoms. The boron layers in MgB_2 are similar to the hexagonal carbon layers on the graph [2]. Unit cell parameters of magnesium boride are a = 3,086 Å and c = 3,524 Å. When the simple hexagonal structure of MgB_2 is examined, it can be seen that magnesium is located at the corners of the structure at the upper and lower surface centers, and boron has a planar structure in the volume center. The bond length values were found to be 0.25017 nm for the Mg-B bond and 0.17909 nm for the B-B bond [5]. Molecular formula of magnesium boride is MgB_2, molar mass 45.93 g/mol, density of 2.6 g/cm^3 and melting point of 1300 °C [6]. For the first time, an intermetallic superconductor has a high critical temperature at 39 °K has intensified interest in MgB_2. The critical temperature of MgB_2 at 39 °K (Transition temperature = transition temperature for superconductivity) offers a higher operating temperature than Nb-Ti (9 °K) and Nb_3Sn (18 °K) superconductors still used in superconductivity applications [7]. It has the highest transition temperature in all intermetallic compounds and in low temperature superconductors. The low atomic mass of the boron atoms is the cause of the high transition temperature. Since these atoms have a higher vibration frequency of 18, they cause the transition temperature to be higher [8].

Studies of MgB_2 superconductivity on various physical properties and superconductivity mechanisms have found that material shows high critical current density (Jc) and high trapped magnetic field (Hc) at low temperatures. Critical magnetic field and critical current density measurements show that magnesium boron is a second type superconductor [9]. The high critical temperature value in MgB_2 gives the hope that higher critical temperatures can be achieved in simple compounds. With the discovery of MgB_2, interest in the superconductivity of nonoxidized compounds has been revitalized (studies on boron compounds), research has begun on superconductivity in boron-related compounds, and several compounds have been announced as superconducting: TaB_2, $BeB_{2.75}$ [10]-[14]. After the discovery of the MgB_2 superconductor, metallic boron layers play a crucial role in the MgB_2 superconductor, while higher element Tc critical temperatures have been predicted for compounds with light elements [15]. MgB_2, magnesium and boron oxide has fewer elements than superconductors. Copper oxide is relatively easier and cheaper to synthesize because it is formed from less unprocessed material than high temperature superconductors [8]. Studies to measure the stability of the MgB_2 compound to atmospheric conditions have shown that the material exhibits very strong hygroscopic behavior. Water and humid air affect MgB_2, even at room temperature, $Mg(OH)_2$, $MgCO_3$ and B_2O_3 [16].

Magnesium boride (MgB_2) has similar properties to Nb_3Sn, which is widely used in superconductivity applications; the higher transition temperature, lower density, and the abundance of both magnesium and boron in nature make it an interesting material for technology. Since magnesium and boron containing compound exhibits superconductivity, it can be performed in all superconductivity applications, such as; electric-electronics and transportation industry, making of strong magnets, and so on. There are many areas available.

Gülşah Çelik Gül is with the Balıkesir University, Turkey (e-mail: gulsahcelik9@gmail.com).

The critical temperature (39 ° K) of MgB_2 is higher than that of metallic superconductors and lower than Cu-O containing high-temperature superconductors discovered in 1986 is a limiting factor in practice. On the other hand, recent developments in low cooling "croyo-cooler" technology have shifted the use areas of MgB_2 to microelectronics and device construction technology [7]. The crucial point of this work is an oxygen-free medium in a domestic microwave oven which constructed by our group has an ability about passing through a gas in the oven to synthesis c-BN, B_4C, MgB_2 etc.

II. Experimental Details

The starting compounds were supplied by Merck Company as analytically pure. Amorphous boron and magnesium source (magnesium nitrate, magnesium stripe wire, and magnesium oxide) were measured 2:1 molar ratio, after several groundings the mixture is transferred into a porcelain crucible. The mixture was irritated to 800 W microwave powers for 30 minutes under pure nitrogen atmosphere. The nitrogen atmosphere was used to remove undesired oxygen in the oven medium. The gas has been passed through the system for 20 minutes to ensure that oxygen is completely removed from the environment before microwave radiation. A gas trap has been added to the end of system to prevent gas leaks back.

In Fig. 1, the close system painted by 3D CAD Design Software SOLIDWORKS is designed by our group. The original photos of the system was drawn same as original structure (Fig. 2). The aim of designing of our oxygen-free glass system is constitution to remove the oxygen from the oven's medium to obtain the highest boron containing boroxide by microwave method. In this way, there are two advantages in same place first is maximum boron containing material last is inactive atmosphere. The glass part of the system was constructed by boroglass which is resisted against sudden temperature rise and connection tubing was made by cupper pipe in oven, and plastic pipes at outside. Microwave radiation is ensured in a domestic microwave oven. Airtightness provided by air trap and alumina tape. Ultra-pure nitrogen was used to remove the oxygen from the medium [17]. The nitrogen flow rate is fixed 3 mL/min at 5 atm pressure. Siemens V12 domestic microwave oven was used to obtain microwave radiation.

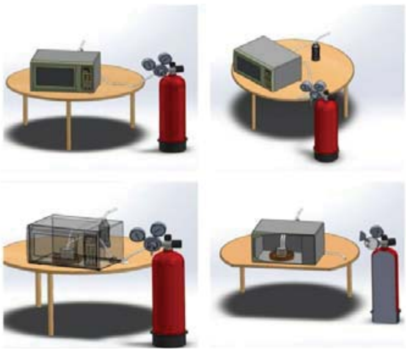

Fig. 1 The closed oxygen-free glass system which designed personally [17]

Fig. 2 Original photos of the closed oxygen-free glass system [17]

The powder XRD pattern for structural characterization was completed by Panalytical X'Pert Pro Diffractometer and CuKα radiation (λ=1.54056Å, 40 mA, 50kV). Perkin Elmer Spectrum 100 FTIR Spectrometer was used to record infrared spectra in the range 4000 and 600 cm^{-1}.

III. Results and Discussion

In Fig. 3, the XRD pattern of the sample was displayed. After the comparison to powder diffraction databank, the results confirm that the patterns correspond to both MgB_4 and MgB_2 compounds. MgB_2 is crystallized in hexagonal crystal system with unit cell parameters a=3.083 Å and c=3.521 Å. On the other hand, MgB_4 is crystallized in orthorhombic crystal system with unit cell parameters a=5.464 Å, b=4.428 Å and c=7.472 Å. Because of the amorphous character of the compound, phase determination seems not possible with only powder X-ray pattern. The relatively close diffraction data do not allow for clear separation of phases. Although all, our aim of obtaining oxygen free magnesium and boron containing compound has been achieved by this personally designed glass system.

In Fig. 4 and Table I, the FTIR spectrum and vibrational data of the sample was given respectively. The wave numbers at the ranges 1400-1500, 900-1000 and 650-750 cm^{-1} are corresponded to various sub-vibrations of boron-oxygen bonds [18].

IV. Conclusion

Boron and magnesium containing compound without oxygen has been synthesized via personally designed close glass system which has a microwave oven, pure nitrogen atmosphere in a glass system and a gas trap. This personally designed glass system has been designed to aim get rid of undesired oxygen which generate structural defects and low specification material. The powder XRD pattern correspond to two magnesium boron containing compounds MgB_4 and MgB_2. FTIR spectrum has been confirmed the boron-oxygen bond to verified the crystal structure.

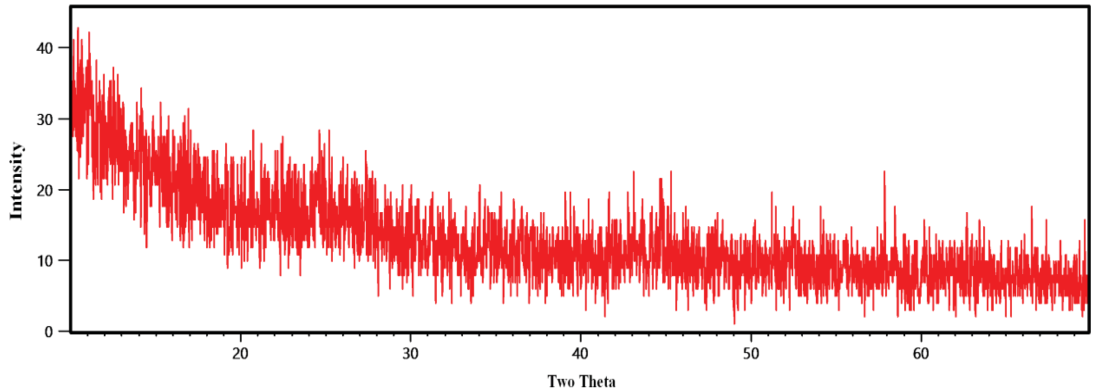

Fig. 3 The XRD pattern of the sample

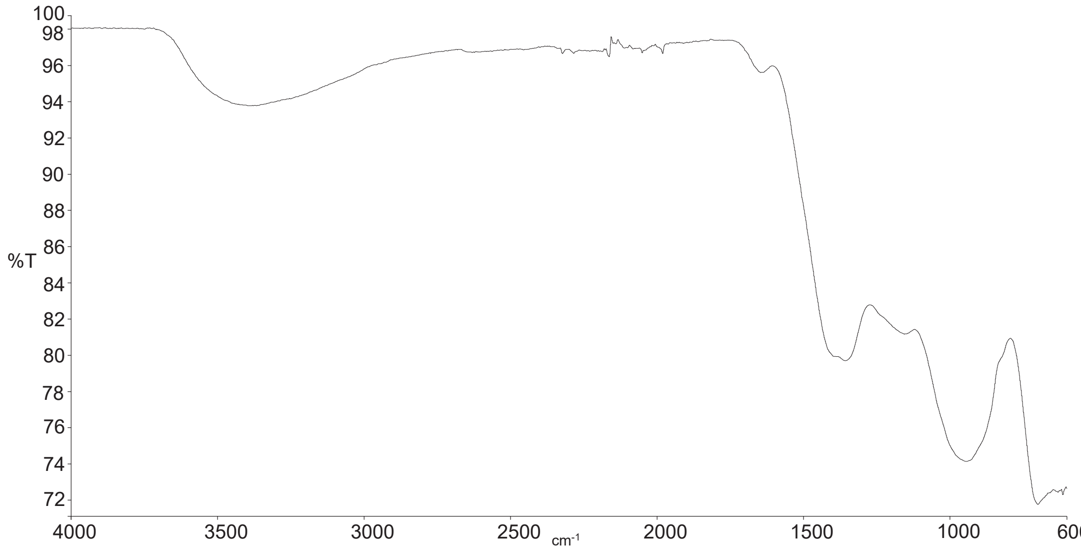

Fig. 4 The FTIR spectrum of the sample

TABLE I
THE VIBRATIONAL DATA OF THE SAMPLE

Assignment	Wave number (cm^{-1})
ν_{as} (B –O)	1400-1500
ν_s (B –O)	900-1000
δ (B –O)	650-750

ACKNOWLEDGMENT

We thank to TUBITAK-BIDEB and BAU-Scientific Research Project Unit for their supports.

REFERENCES

[1] Jones, M. E. and Marsh R. E. (1954). "The Preparation and Structure of Magnesium Boride, MgB2". Journal of the American Chemical Society 76: 1434–1436.
[2] Nagamatsu, J., Nakagawa N., Muranaka T., Zenitani Y., Akimitsu J., 2001, "Superconductivity at 39 K in MgB2", Nature 410, 63.
[3] Buzea, C; Yamashita, T; 2001, Review of Superconductor Proerties of MgB2, CondMat/0108265 to Appear Superconductors, Science and Technology, Topical Review.
[4] King, R.B., 2002. The Similarities Between Magnesium Diboride and Cuprate Superconductors and the Role of Subvalent Magnesium, Polyhedron, 21, 2347– 2350.
[5] Hua, H. L., et all. , 2001, Chin. Phys. Soc. and IOP Publishing Ltd., 104.
[6] Larbalestier, D C, Rogado N, Regan K A, Hayward M A, He T, Slusky J S, Inumaru K, Haas M K and Cava R J, 2001, Thin Film Magnesium Boride Superconductor with Very High Critical Current Density and Enhanced Đrreversibility Field, Nature 411 558.
[7] Mustafa Zafer Balbağ, Production of Magnesıum, Boron and Magnesıum Diboride Thin Films Using Thermionic Vacuum Arc (TVA) Technique and Investigation of Some Physical Properties, PhD thesis, Eskişehir Osmangazi Üniversitesi Fen Bilimleri Enstitüsü, 2009.
[8] Savaşkan, B., 2007, Production of MgB2 and investigation of some properties, PhD thesis, Karadeniz Teknik Üniversitesi Fen Bilimleri Enstitüsü, 93 s.
[9] Buzea, C; Yamashita, T; 2001, Review of Superconductor Properties of MgB2, Cond Mat/0108265 to Appear Superconductors, Science and Technology, Topical Review.
[10] Fenler, I., 2001, Absence of Superconductivity in BeB2, Physica C, 353, 11.
[11] Young, D, P; Adams P, W; Chan C, Y; and Fronzek F, R; 2001, Preprint, Structure and Superconducting Properties of BeB2, cond-

mat/0104063.
[12] Gasparov, V. A., Sidorov N, S., Zver'kova I, I., and Kulakov M, P., 2001, Electron Transport in Diborides: Observation of Superconductivity in ZrB2, JETP Lett. 73, 532.
[13] Kaczorowski, D., Zaleski A, J., Zogal O, J., and Klamut J., 2001, Preprint, Incipient Superconductivity in TaB, Cond-mat/0103571.
[14] Strukova, G, K., Degtyareva V, F., Shovkun D, V., Zverev V, N., Kiiko V, M., Ionov A, M., and Chaika A, N., 2001, Preprint, Superconductivity in the Re-B System, Cond-mat/0105293.
[15] Kortus, J., MazinI, I., Belashcenko K., D; Antropov V, P., and Boyer L, L., 2001, "Superconductivity of metalic boron in MgB2" Phys. Rev. Lett. 86, 4656.
[16] Sen, S., Aswal D., Sıng A., 2002. Preparation and Characterization of MgB2 Superconductor, Journal of Physics, 867-870.
[17] Çelik Gül, G., Kurtuluş, F., 2015, "Pioneer synthesis and characterization of boron containing hard materials" International Journal of Chemical, Molecular, Nuclear, Materials and Metallurgical Engineering 9, 9.
[18] Azmi Seyhun KIPCAK, Synthetic production of magnesium borates from different magnesium and boron sources by various methods and the investigation of their production parameters, PhD thesis, Yıldız Technical University Graduate School of Natural and Applied Sciences, 2013.

Gülşah Çelik Gül was born in Balıkesir, Turkey on 9th of July in 1986. She graduated from University of Uludağ, Department of Chemistry in 2008. She has a master degree of science and PhD degrees in chemistry from Balıkesir University. Her major field is inorganic chemistry.

She works as a Project Expert at Balıkesir University since 2013. She was published eleven articles in SCI-expanded journals related with inorganic chemistry and bioinorganic chemistry. Her current and previous research interests are to material science, bioinorganic chemistry, solid state chemistry, crystallography and powder X-ray diffraction. Dr. Çelik Gül, is a member of Turkish Chemistry Society and Turkish Biochemical Society.

Figen Kurtuluş was born in Balıkesir, Turkey in 1972. She graduated from University of Uludağ Department of Chemistry Education in 1993. She has a master degree of science and PhD in chemistry from Balıkesir University. Her major field is inorganic chemistry.

She has been Prof. Dr at Balıkesir University Chemistry Department since 2016. She was published twenty articles in SCI-expanded journals related with inorganic chemistry and solid-state chemistry. Her current and previous research interests are to inorganic chemistry, material science, solid state chemistry, crystallography and powder X-ray diffraction.Prof. Dr. Kurtuluş is a member of Turkish Chemistry Society.