Proceedings of ICXRI 2010
International Conference on X-Rays & Related Techniques in Research & Industry
June 9 – 10, 2010, Aseania Resort Langkawi, Malaysia

ICXRI 2010

Formation of TiC-Reinforced Iron Based Composite Through Carbothermal Reduction of Hematite and Anatase

Mohd Salihin Hassin*, Zuhailawati Hussain and Samayamutthirian Palaniandy
School of Materials and Mineral Resources Engineering,
Engineering Campus, Universiti Sains Malaysia,
14300 Nibong Tebal, Penang, Malaysia
*email: salihin212@gmail.com, phone: 604-5995258, fax: 604-5941011

Abstract— In this research carbothermal reduction of mechanical activated hematite (Fe_2O_3), anatase (TiO_2) and graphite (C) was investigated. The mixtures of raw materials with composition of Fe-20vol%TiC were mechanically activated in a planetary ball mill with different milling time (0h-60h) in argon atmosphere. X-ray diffraction (XRD) results showed the intensity of Fe_2O_3 reduce with milling time. The activated powders were pressed using cold press under constant pressure (100MPa) and heat treated at 1100°C for sintering temperatures in vacuum furnace. The increase in milling time resulted in the formation of iron (Fe) and titanium carbide (TiC) phase as confirmed by XRD result.

Keywords: Mechanical activation, carbothermal reduction, milling time, sintering temperature

I. INTRODUCTION

Iron and iron alloys are common metals and the ferromagnetic materials in everyday use. According to Pagounis et al., [1] reported iron and its alloy as a matrix in material in composite system is very interesting based on their low cost, the possibility of heat treatment and technological effectiveness. Basically iron metal is produced from iron ore or hematite (Fe_2O_3) according to opportunity to use relatively cheap and abundant raw materials [2]. Iron can be produced by reduction of iron oxide with several syntheses such as carbothermal reduction or mechanochemical processing, thermal plasma synthesis, and self-propagating high temperature synthesis (SHS) [3].

Titanium carbide (TiC) has desirable properties as the reinforcing phase in composite. TiC is one of the most suitable reinforcement in iron composite due to its higher hardness and high chemical stability and also can be wetted by iron matrix [4].

In recent years, carbothermal reduction via mechanical activation (MA) or known as mechanochemical processing (MCP) has been used to produce advanced material. Carbothermal reduction of the mineral ilmenite ($FeTiO_3$) with carbon under argon or vacuum has resulted in the formation of a fine powder consisting of iron metal and titanium carbide [5]. The reductions of iron oxides by carbonaceous materials have been carried out on mechanical activation (MA) of iron oxides and their mixtures with reductant materials [6].

In this study, iron based as raw material is chosen because it offers the opportunity to use relatively cheap and abundant raw materials without compromise the required properties of advanced material by composited with TiC reinforced.

II. EXPERIMENTAL PROCEDURE

Hematite (Fe_2O_3), titanium oxide (TiO_2) and graphite (C) were used as raw materials. The mixtures of raw materials which corresponds Fe-20vol%TiC were milled by using a high-energy Fritsch Pulverisette P-5 planetary mill in various milling time i.e. 0h (non-milled), 10h, 20h, 30h, 40h and 60h. The weight ratio of ball to powder was 10:1 in all experiments were use .The milling process was done in argon atmosphere.XRD analysis was carried out in order to detect phase formation in the as-milled powder. The as-milled powder was compacted under 100MPa of pressure by using cold press machine to consolidate the as-milled powder. The pallet was sintered in a vacuum furnace at 1100°C of sintering temperature and then cooled in furnace to room temperature. The soaking time was 1 h. The sintered pallet was analyzed by X-Ray diffraction (XRD) analysis to evaluate the formation of TiC in iron matrix after carbothermal reduction of oxide material.

III. RESULTS & DISCUSSION

XRD Analysis

XRD patterns of the sample containing Fe_2O_3, TiO_2 and C which was milled for 0-60 h with time interval of 10 h are shown in Figure 1. The results show that without milling (0 h), peaks of Fe_2O_3, TiO_2 and C are clearly detected. After milling (10 h-40 h), the intensity of all

peaks reduced with increasing milling time. There is also a shift in the position of all the diffraction peaks due to milling. Compared with the XRD pattern taken from the same composition without milling (0 h) and after milling, the graphite and TiO_2 peaks were dramatically reduced when milling started. Therefore graphite structure is easily deformed during mechanical activation. Graphite peaks completely disappeared after 10 h milling time is in agreement with the results of previous study [6]. All the peaks of as-milled powder were continuing decrease in intensity and increase in broadening by increasing milling time from 10 h to 40 h. But at the longer milling time (60 h), phase transformation occurred whereby hematite ($Fe_2O_{3)}$ was completely transformed to magnetite (Fe_30_4).

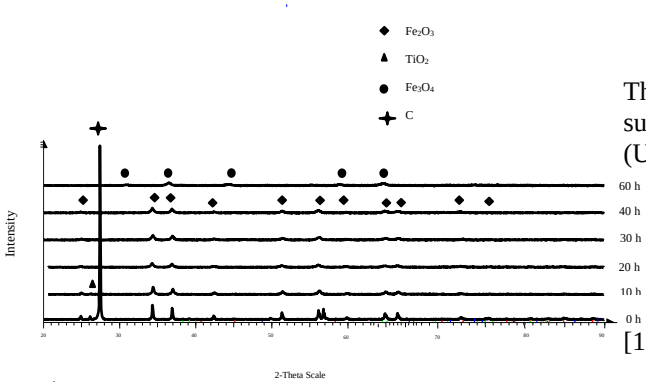

Figure 1: XRD patterns of Fe_2O_3-TiO_2 -C powder as-milled with milling times of 0 h, 10 h, 20 h, 30 h, 40 h and 60 h

Figure 2 shows XRD patterns of sintered Fe–TiC compact at different milling times at 1100°C. It was observed that Fe diffraction peaks slightly broadened with increasing milling time after sintering. At 0 h of milling time, Fe peaks were detected but TiC peaks were not detected but after milling in 10 h and above, both Fe and TiC phases were identified. However the broadening of Fe and TiC peaks of 10 h until 40 h was slightly same. This result suggests that without applying heat, the internal energy of the particles obtained during mechanical activation at is not enough to initiate the reaction to form Fe and TiC phase [7]. At 0 h, phase of Ti_2O_3 was detected.

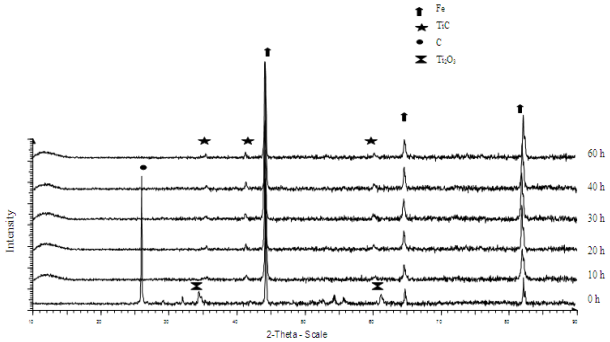

Figure 2: XRD patterns of sintered Fe-TiC with milling times of 0 h, 10 h, 20 h, 30 h, 40 h and 60 h at 1100°C

IV. CONCLUSION

The XRD diffraction pattern showed that the hematite (Fe_2O_3) phase in the as-milled powder was broadened with increasing milling time (0h-60h). Magnetite peaks appeared in XRD patterns of the entire sample milled for 60 h. It has been indicated that some of hematite particles have been reduced to magnetite due to milling in the presence of graphite. Sintering at 1100 °C of the Fe_2O_3-TiO_2-C compact transform the phase to Fe-TiC composite and enhanced the intensity of the Fe peaks in XRD patterns.

ACKNOWLEDGEMENT

The authors are pleased to acknowledge the financial support for this research by Universiti Sains Malaysia (USM) Fellowship and research grant FGRS (6071194).

REFERENCES

[1] E. Pagounis, M. Talvitie, and V. K. Lindroos. 'Influence of the Metal/Ceramic Interface on the Microstructure and Mechanical Properties of Hiped Iron- Based Composites', *Composites Science and Technology* (1996), 56, 1329-37

[2] I. W. M. Brown, and W. R. Owers. 'Fabrication, Microstructure and Properties of Fe-Tic Ceramic-Metal Composites', *Current Applied Physics* (2004), 4, 171-4

[3] Suryanarayana, C. (2003). Mechanical Alloying and Milling, Marcel Dekker, US, p.35-122, 269-270, 359, 385-399

[4] Nuilek, K., Memongkol, N., and Niyomwas, S. 'Production of titanium carbide from ilmenite', *Songklanakarin J. Sci. Technol* (2008). 30 (2), 239-242

[5] Ian W. M. Brown. 'Advanced Ceramic Research at Industrial Research Limited', *Advanced Engineering Materials* 2002, p.58-59.

[6] R. Tahmasebi, M. Shamanian, M. H. Abbasi, and M. Panjepour. 'Effect of Iron on Mechanical Activation and Structural Evolution of Hematite-Graphite Mixture', *Journal of Alloys and Compounds* (2009), 472, 334-42.

[7] H. Zuhailawati, H.M. Salihin, and Y. Mahani. 'Microstructure and properties of copper composite containing in situ NbC reinforcement: Effects of milling speed', *Journal of Alloys and Compounds* 489 (2010) 369–374