UNIVERSITI SAINS MALAYSIA

Laporan Akhir Projek Penyelidikan Jangka Pendek

Carbothermal Reduction of Mechanical Activated Hematite and Anatese Mixture for Synthesis of Iron Based Composite

by
Dr. Zuhailawati Hussain
Dr. Samayamutthirian Palaniandy

2012

F0480

BORANG FRGS – P3(R)

FINAL REPORT
FUNDAMENTAL RESEARCH GRANT SCHEME (FRGS)
Laporan Akhir Skim Geran Penyelidikan Asas (FRGS) IPT
Pindaan 1/2010

A

RESEARCH TITLE : **Fabrication of TiC-reinforced Iron Based Composite Through Carbothermal Reduction and Mechanical Activation of Hematite and Anatase**
Tajuk Penyelidikan

PROJECT LEADER : DR. ZUHAILAWATI HUSSAIN
Ketua Projek

PROJECT MEMBERS (including GRA) : 1. DR. SAMAYAMUTTHIRIAN PALANIANDY
2.
Ahli Projek

PROJECT ACHIEVEMENT *(Prestasi Projek)*

B

ACHIEVEMENT PERCENTAGE			
Project progress according to milestones achieved up to this period	**0 - 50%**	**51 - 75%**	**76 - 100%**
Percentage			**100%**

RESEARCH OUTPUT

	Indexed Journal	*Non-Indexed Journal*
Number of articles/ manuscripts/ books *(Please attach the First Page of Publication)*	1. Mohd Salihin Hassin, **Zuhailawati Hussain** and Samayamutthirian Palaniandy (2011) Formation of TiC-Reinforced Iron Based Composite Through Carbothermal Reduction of Hematite and Anatase **Advanced Materials Research** Vol. 173 (2011) pp. 116-121 2. Mohd Salihin Hassin, **Zuhailawati Hussain** and Samayamutthirian Palaniandy (2011) Sintering of Fe-TiC composite Prepared by Carbothermal Reduction of Hematite and Anatase **Key Engineering Materials** Vols. 471-472 (2011) pp 670-673	

Conference Proceeding *(Please attach the First Page of Publication)*	**International**	***National***
	1. Mohd Salihin Hassin, Zuhailawati Hussain and Samayamutthirian Palaniandy (2010) Formation of TiC-Reinforced Iron Based Composite Through Carbothermal Reduction of Hematite and Anatase **International Conference on X-Rays and Related Techniques in Research and Industry (ICXRI-2010)**, 9-10 June 2010, Langkawi, Malaysia. 2. Mohd Salihin Hassin, Zuhailawati Hussain and Samayamutthirian Palaniandy (2011) Sintering of Fe-TiC composite Prepared by Carbothermal Reduction of Hematite and Anatase **Eighth International Conference on Composite Science and Technology**, March 22 – 24, 2011 at Kuala Lumpur, Malaysia.	
Intellectual Property *(Please specify)*		

HUMAN CAPITAL DEVELOPMENT

Human Capital	**Number**				**Others** (please specify)
	On-going		**Graduated**		
Citizen	Malaysian	Non Malaysian	Malaysian	Non Malaysian	
PhD Student					
Master Student	2				
Undergraduate Student	1				
Total					

EXPENDITURE *(Perbelanjaan)*

C

Budget Approved *(Peruntukan diluluskan)* : **RM 24,000**
Amount Spent *(Jumlah Perbelanjaan)* : **RM 23,9351**

Balance *(Baki)* : RM64.90
Percentage of Amount Spent : 99.7 %
(Peratusan Belanja)

D ADDITIONAL RESEARCH ACTIVITIES THAT CONTRIBUTE TOWARDS DEVELOPING SOFT AND HARD SKILLS

(Aktiviti Penyelidikan Sampingan yang menyumbang kepada pembangunan kemahiran insaniah)

International

Activity	Date (Month, Year)	Organizer
(e.g : Course/ Seminar/ Symposium/ Conference/ Workshop/ Site Visit)		

National

Activity	Date (Month, Year)	Organizer
(e.g : Course/ Seminar/ Symposium/ Conference/ Workshop/ Site Visit)		

E PROBLEMS / CONSTRAINTS IF ANY *(Masalah/ Kekangan sekiranya ada)*

Not much analysis related to thermodynamic study have been conducted which need to be carried out in SIRIM since grant amount is small

F RECOMMENDATION (*Cadangan Penambahbaikan*)

RESEARCH ABSTRACT – Not More Than 200 Words *(Abstrak Penyelidikan – Tidak Melebihi 200 patah perkataan)*

Investigations on mechanical activation of in situ composite have mostly focused on synthesizing composite powder from direct raw material (Fe-TiC) or carbonaceous material such as ilmenite ($FeTiO_3$). In literature, little information is available regarding the effect of processing parameter on properties of Fe-TiC composite from reduction of mineral hematite (Fe_2O_3) with anatase (TiO_2). Therefore, the present work aims to reveal the relationship between processing parameters such as milling time and sintering parameters of Fe-TiC composite with the microstructure and mechanical properties of Fe-TiC composite. 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 (Fe_3O_4) peaks appeared in XRD pattern sample milled for 60 h which indicated that some of hematite particles have been reduced to magnetite due to milling in the presence of graphite. Mechanical activated sintering at 1100 °C of the Fe_2O_3- TiO_2-C compact transformed the phases to Fe-TiC composite with enhanced intensity of Fe peaks in XRD patterns. The formation of Fe-TiC composite was also confirmed by SEM examination and EDX analysis.

Date : 5 April 2011
Tarikh

Project Leader's Signature:
Tandatangan Ketua Projek

COMMENTS, IF ANY/ ENDORSEMENT BY RESEARCH MANAGEMENT CENTER (RMC)
(Komen, sekiranya ada/ Pengesahan oleh Pusat Pengurusan Penyelidikan)

F

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.....

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Name:
Nama:

Signature:
Tandatangan:

Date:
Tarikh:

UNIVERSITI SAINS MALAYSIA

FINAL REPORT OF FUNDAMENTAL RESEARCH GRANT SCHEME (FRGS)

CARBOTHERMAL REDUCTION OF MECHANICAL ACTIVATED HEMATITE AND ANATASE MIXTURE FOR SYNTHESIS OF IRON BASED COMPOSITE

Project Number: 6071174

Duration: 1 MAY 2010 – 31 APRIL 2012

Project Leader: Dr. Zuhailawati Hussain

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Abstract

This study investigated the influence of milling on the formation of TiC-reinforced iron composite through carbothermal reduction of hematite and anatase mixture. A mixtures of hematite, anatase and graphite powders were mechanically activated in a planetary ball mill in an argon atmosphere with different milling times (0-60h). X-ray diffraction showed that with increasing milling time, crystallite size of hematite decreased to nanometer range accompanied by an increment in internal strain. Prolonging the milling process increased dislocation density of the as-milled powder. The as-milled powder was consolidated by cold pressing under 100MPa and sintered in vacuum at 1100°C. High temperature during sintering resulted in the formation of iron and titanium carbide phases as confirmed by X-ray diffraction, scanning electron microscope and energy dispersive X-ray analysis. Without mechanically activated milling, the reaction forming TiC did not occur during sintering at 1100°C, indicating a reduction in reaction temperature promoted by mechanical milling. An increase in milling time resulted in an increase in sintered density and hardness due to the fineness of the composite powder, together with complete TiC and iron phase formation.

Keywords: Hematite; Anatase; Carbothermal reduction; Milling time; Fe-TiC composite

1. Introduction

Industrial development of iron matrix composites has attracted considerable interest due to the composites' advantages in terms of its usefulness in production of inexpensive wear-resistant parts, the possibility of improving its properties through heat treatment and its suitability to be fabricated using various methods including powder metallurgy, conventional melting and carbothermal reduction [1-2]. Typically, an iron matrix is reinforced with hard compounds of ceramic particles such as Al_2O_3, Cr_3C_2 and TiC. With such desirable

properties as improved hardness and high chemical stability, TiC is the most suitable compound for reinforcement of a soft iron matrix. Furthermore, iron and TiC wet each other very well [3]. In comparison with tungsten carbide (WC), TiC has 33% higher hardness, a lower density and higher thermal stability [4]. Thus, TiC can be used in the manufacture of wear-resistant parts, cutting tools, grinding wheels, high temperature heat exchangers, magnetic record heads, turbine engine seals, and electrode or coating materials [3].

Iron is typically synthesized via extraction or reduction of iron ores through such techniques such as mechanochemical processing or carbothermal reduction, thermal plasma synthesis and self-propagating high temperature synthesis (SHS) [5]. Carbothermal reduction via mechanical activation (MA) has been used to produce advanced materials such as synthesized oxide material for extraction to produce metallic material. Thus, this method has been comprehensively studied in the fields of extractive metallurgy, materials synthesis and production of nanocrystalline and amorphous materials [6].

Collision energy during mechanical milling activates a chemical reaction by decreasing the activation temperature. Thus, this process has been used in mineral processing to produce finely ground particles, to increase surface area and to improve chemical reactivity of the milled product [6]. The fraction of milling energy transferred to the powder mixture may affect the properties of the powder by increasing crystal defects such as dislocation, structural distortion, and atom displacement as well as by the formation of amorphous phase [7]. Thus, during mechanical milling chemical reactions could occur at particle contact areas, changing the composition of the powder mixture.

Mechanical activation has been used to reduce iron oxide with reductant materials such as graphite in order to produce iron powder [7]. According to Brown and Owers [8], synthesis of metal powder using abundant, relatively inexpensive oxide raw materials is

promising. For example, Brown [9] has reported reduction of ilmenite ($FeTiO_3$) by adding carbon to produce a fine powder of iron-titanium carbide mixture under argon or vacuum atmosphere. Anatase (TiO_2) has also been reduced with carbon to produce TiC [5, 10]. However, these studies have dealt with carbothermal reduction of single metal oxide to produce iron, TiC or TiC-reinforced iron composite [5, 10]. Carbothermal reduction of metal oxide mixtures to produce TiC-reinforced iron composite has not yet been reported.

In this study, instead of ilmenite ($FeTiO_3$), a mixture of two metal oxides, with hematite (Fe_2O_3) as iron source and anatase (TiO_2) as titanium source, was chosen for reduction with graphite to produce a Fe-TiC composite. These materials were selected to take advantage of the relative cheapness and abundance of raw hematite while compromising on the required properties of an iron-based composite. The reduction of the mineral hematite (Fe_2O_3) with anatase (TiO_2) and graphite (C) results in the formation of a fine powder consisting of iron metal and titanium carbide, as summarized in Eq. 1:

$$Fe_2O_3 + TiO_2 + 6C \rightarrow 2Fe + TiC + 5\,CO \qquad (1)$$

This study investigated the effectiveness of carbothermal reduction of hematite and anatase with aid of a mechanical milling process. Density and hardness of the Fe-TiC composite were correlated with the microstructure of the obtained composite.

2. Experimental Procedure

A mixture of hematite (Fe_2O_3;95% pure, <10μm), anatase (TiO_2;99% pure, <0.16 μm) and graphite (C;99.8% pure, <0.1 μm) powders with composition corresponding to Fe-20vol%TiC was milled in a high-energy Fritsch Pulverisette P-5 planetary mill for various milling times, i.e. 0h (mixed powder), 10h, 20h, 30h, 40h and 60h. The ball to powder ratio by weight was10:1, and stainless steel balls of similar diameter (20 mm) were used. Phase

formation of activated powder was analyzed by x-ray diffraction (XRD). The particle size of the activated powder was measured by Mastersizer particle size analysis. As-milled powder was consolidated using cold pressing at a constant pressure of 100 MPa. Heat treatment for sintering and carbothermal reduction was performed in a vacuum furnace for 1 hour soaking time at a temperature of 1100°C. The sintered pellet was analyzed by XRD to investigate the formation of TiC in the iron matrix after carbothermal reduction of metal oxides. Crystallite size and internal strain of hematite (Fe_2O_3) were calculated using two of the highest intensity XRD peaks of hematite via the Williamson-Hall method, as shown in the following equation [11]:

$$B_r \cos\theta = \frac{0.89\lambda}{D} + 2\eta Sin\theta \qquad (2)$$

where θ is the Bragg angle, D is the average crystallite size, B_r is the line broadening, λ is the X-ray wavelength and η is the internal strain. The microstructures of the as-milled powder and the sintered Fe-TiC composite were observed under scanning electron microscopy (SEM) in secondary electron and backscattered electron modes. Formation of iron matrix and TiC reinforcement phases was confirmed by energy dispersive X-ray (EDX) and XRD analyses. Archimedes' principle was used to measure sintered density whilst the Vickers microhardness test was used with a load of 0.5 kg and 10 s dwell time to measure the hardness of the composite.

3. Results and Discussion

3.1 XRD Analysis

Fig. 1 shows the XRD patterns of activated powder mixtures of Fe_2O_3, TiO_2 and C which had been milled for 0 h-60 h. The un-milled sample (0 h) shows sharp Fe_2O_3, TiO_2 and C peaks since these three were the main starting powders. Broadening of all the peaks was observed when mechanical activation began. With milling for 10 h-40 h, the intensity of all peaks reduced with increasing milling time. In particular, the height of the graphite and TiO_2 peaks dramatically declined once milling was started, indicating the refinement of as-milled powder and the formation of amorphous structure. Schaffer and Forrester [12] have reported that during ball to powder collision, the milling energy exerts a greater effect on graphite particles than on hematite (Fe_2O_3) since graphite is softer than hematite and therefore its structure is easily deformed and transformed into an amorphous state during ball milling. The disappearance of graphite peaks was similarly found in a previous study [7] when hematite and graphite were activated. The reduction of intensity with the broadening of Fe_2O_3, TiO_2 and C XRD peaks during milling for 10 h to 40 h aligns with the findings of Razavi and Rahimipour [10], who observed that peaks showed a small broadening as well as reduction in intensity as $FeTiO_3$ and C phases became finer.

A sudden phase transformation of hematite (Fe_2O_3) to magnetite (Fe_3O_4) occurred when the mixture of raw materials was milled for 60 h. The appearance of magnetite resulted from the mechanical activation of hematite particles at longer milling time due to reduction of hematite. This finding suggests that sufficient reduction energy was provided by the transferred energy from the impact energy of balls on the as-milled powder during mechanical milling to promote this transformation to magnetite. According to Tahmasebi et al. [7], the energy of activated hematite and graphite particles increased locally due to ball to

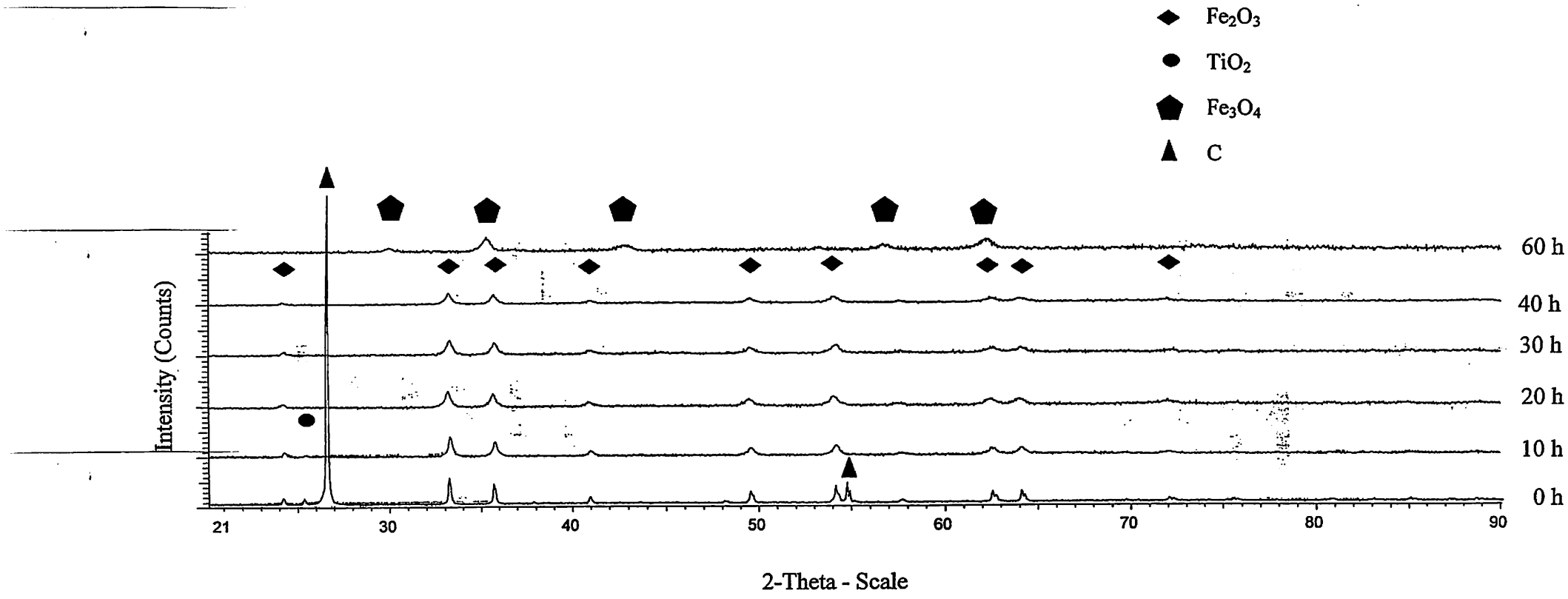


Fig. 1.XRD patterns of Fe_2O_3-TiO_2 -C as-milled powder with different milling times

ball or ball to jar wall impact. They also reported that the presence of graphite in activated hematite facilitated the reduction of hematite to magnetite as a consequence of the presence of fresh and highly excited powder surfaces as particles fractured in the intense and localized impact of milling. A similar result was achieved by Matteazzi and Le Caër [13], who found that the hematite was converted to nanocrystalline magnetite phase with an average crystallite size of 10 nm after 70 h of high energy dry milling under a nitrogen atmosphere. It would be difficult to further reduce an oxide material such as hematite to metallic phase because the stored energy in activated material is not high enough to overcome the endothermic reduction of iron oxide by graphite [7, 13-14].

Using the Williamson-Hall method, the XRD patterns were used to calculate the crystallite size and internal strain of the as-milled powder. Fig. 2 shows the data for crystallite size and internal strain for hematite and magnetite. According to Fig. 2, with increasing milling time, the crystallite size of hematite and magnetite particles decreased while the internal strain increased. The crystallite size of hematite drastically reduced from 89.23 nm (0 h milling) to 50.82 nm (10 h milling) due to considerable fracturing during mechanical milling. The rate of crystallite size reduction decreased with milling for 10 h to 40 h because the level of refinement of powder mixtures appeared to become almost stable with prolonged milling. The wide difference in crystallite sizes of powder milled for 40 h (31.81 nm) and powder milled for 60 h (18.15 nm) is probably caused by the phase transformation from hematite to magnetite.

Fig. 4 shows XRD patterns of consolidated pellets prepared with different milling times and sintered at 1100°C. It was observed that only Fe and TiC peaks present with iron became the major composition in the sintered products prepared from milled powder. For the pellet made of un-milled powder (0 h), Fe peaks were detected but not TiC peaks. However, for pellets formed from powders after10 h milling time and longer, both Fe and TiC phases were identifiable. Fe diffraction peaks slightly broadened with increasing milling time. However, the intensity and broadening of Fe and TiC peaks for milling times of 10 h until 40 h were almost same. Although the peaks of Fe and TiC were clear, the XRD patterns show that the structure was not perfectly crystalline.

Without heat treatment, the formation of titanium carbide-reinforced iron composite did not occur as the heat produced during milling was insufficient. The energy transferred to the powder and the increase in internal energy of the particles during mechanical milling was not enough to initiate the reaction to form Fe and TiC phases. Therefore, the reaction forming Fe and TiC phases only took place during sintering at 1100°C. At 0 h, phase of Ti_2O_3 resulting from the reduction of anatase (TiO_2) was detected. The reaction leading toTiC formation did not happen during heat treatment of non-mechanically-activated powder since the activation energy of the powder was too low. However, at 60 h of milling, the formation of iron matrix was clearly detected in XRD peaks as a result of reduction of magnetite to iron metal, a finding which agrees with that of previous work [6]. It may be that TiC peaks could not be detected because of the high level of refinement of anatase with prolonged milling.

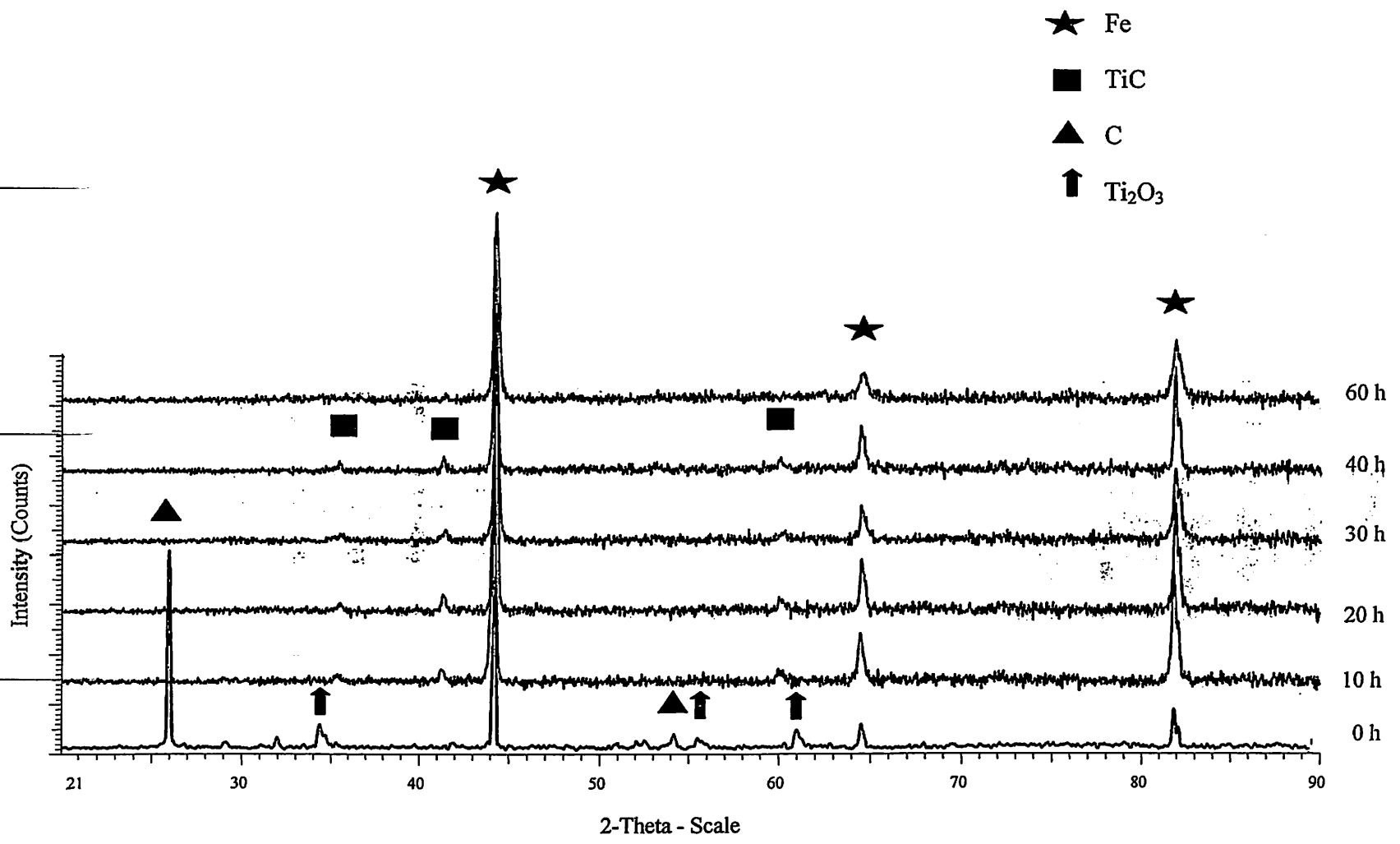


Fig. 4. XRD patterns of Fe-TiC made with different milling times and sintered at 1100°C

Based on reaction (1), an Ellingham-Richardson diagram for the formation of Fe-TiC from oxide metals was plotted (Fig.5). The Ellingham-Richardson diagram shows the lowest temperature allowing the reaction forming Fe-TiC composite to take place is about 900°C (1173K), corresponding to free Gibbs energy ($\Delta G°$) equal to zero. The free Gibbs energy for the equilibrium formation according to reaction (1) can be calculated based on the following equation [15-16]:

$$\Delta G° = 981922.25 - 837.691T \quad (3)$$

where temperature and energy are in terms of Kelvin and Joule, respectively. Fig. 5 shows that sintering the consolidated pellet at 1100°C (1373 K) enabled the reaction to occur and to become endothermic as the free Gibbs energy has a negative value at this temperature. Fig. 5 also shows that the partial pressure of CO, which is affected by continuous transformation of CO and CO_2 gases during the reaction, varied across temperatures. The formation of Fe-TiC composite at 1100°C (1373 K) sintering temperature only successfully occurred when the partial pressure was less than 6.45×10^5 Pa.

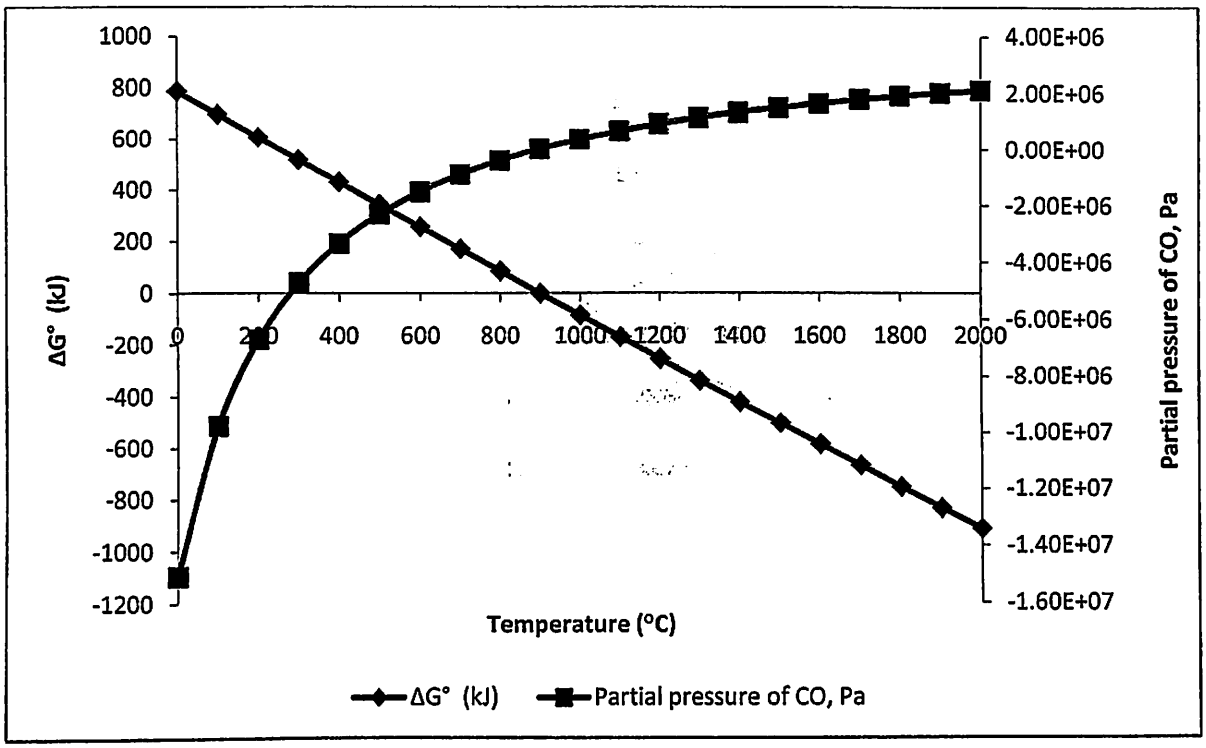


Fig. 5. Ellingham-Richardson diagram and effects of partial pressure of CO on reduction of hematite and anatase

Razavi et al. [4] have found that the formation of TiC phase after carbothermal reduction of activated TiO_2 only successfully occurred after heat treatment at 1250°C. However, results of the present work show that carbothermal reduction of activated TiO_2 and Fe_2O_3 mixture forming Fe-TiC could take place at a temperature as low as 1100°C. This finding suggests that besides acting as the metal matrix for Fe-TiC composite, hematite is important in its role as a catalyst for reducing the carbothermal reduction temperature of TiO_2. This explanation is supported by Setoudeh et al. [17], who observed that addition of free iron caused the carbothermal reduction of anatase to start at a lower temperature than that required with no addition of free iron.

Comparing the XRD patterns of sintered pellets prepared using non-milled and milled Fe_2O_3-TiO_2-C mixtures (Fig.4), TiC phase was present only in the milled pellets, indicating

that the carbothermal reduction of hematite and anatase mixture during sintering at 1100°C only took place in the milled pellet. Thus, it can be concluded that although the sintering temperature was higher than 900°C, which represents the minimum temperature required for the reaction to take place as indicated by the free Gibbs energy calculation, the reaction to form TiC was found not to occur during sintering at 1100°C without prior mechanical milling.

3.2 Microstructure Evolution

The morphology of activated composite powders with various milling times was observed under SEM using secondary electron mode, as shown in Fig. 6. In the un-milled mixture, hematite and graphite particles can be easily recognized because the powders were just blended due to low mixing energy. This confirms the XRD analysis of the un-milled sample, in which peaks of the starting materials (hematite, anatase and graphite) were clearly detected. The flaky shape of graphite was also clearly observed. The morphology of the starting powder changed after mechanical activation, especially in terms of particle sizes and shapes. However, after 60 h milling, agglomeration of powder occurred because fresh and atomically clean surface fractured powders were cold welded during prolonged milling [5].

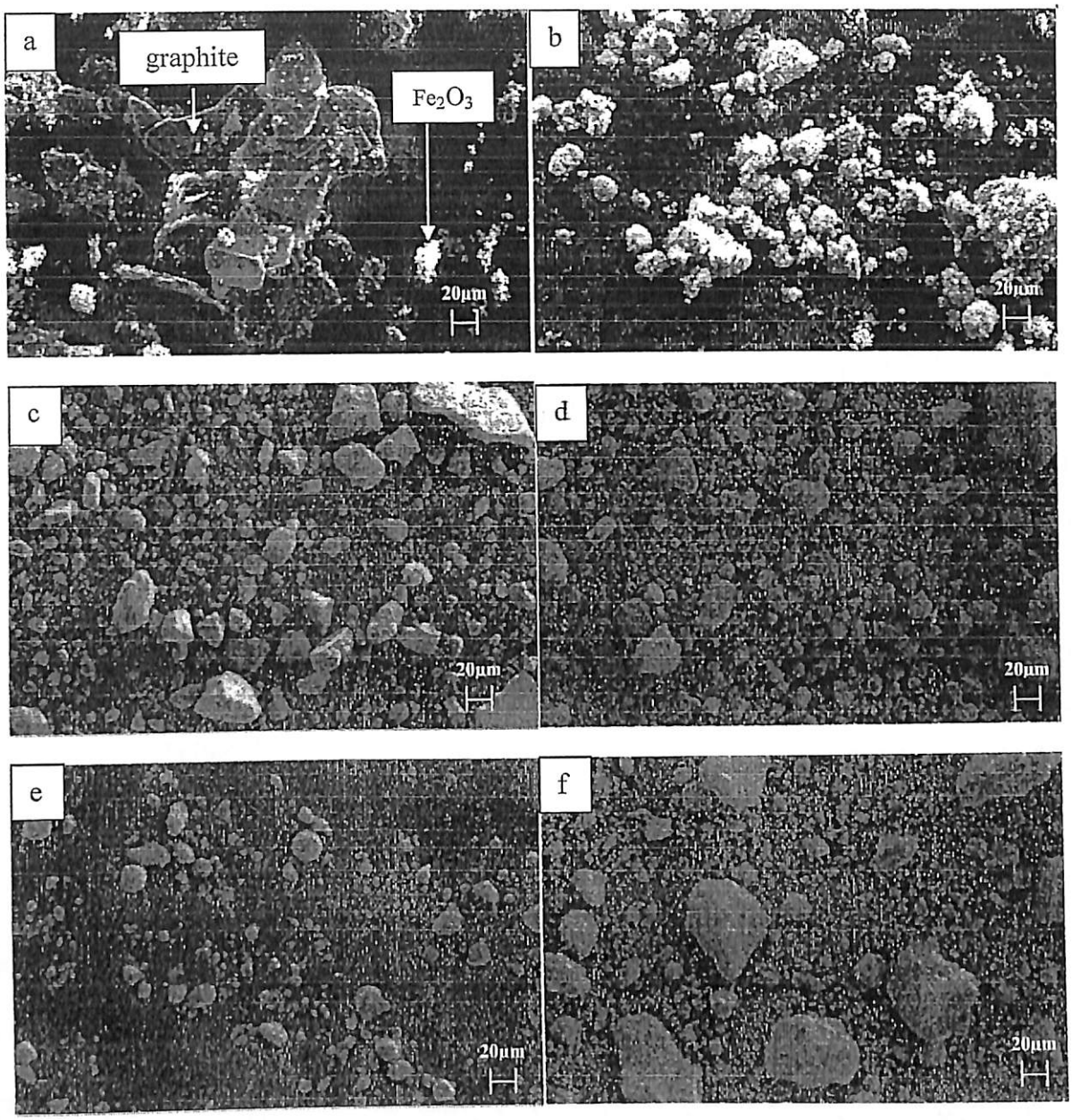


Fig. 6. SEM micrograph of hematite-anatase-graphite with different milling times: (a) 0h, (b) 10 h, (c) 20 h, (d) 30 h, (e) 40 h and (f) 60 h

Fig. 7 shows the SEM (backscattered electron mode) image of the microstructure of sintered Fe-TiC prepared using powders milled for 10 h to 60 h. The microstructure shows distribution of enriched titanium carbide (grey area) within the iron matrix (white area). For powders milled up to 40 h, the microstructures of the composites were very similar except that the carbide was more uniformly distributed in the iron matrix with increasing milling time. In the EDX image in Fig. 8 the enriched TiC phase is seen embedded homogenously in

the iron matrix. It is suggested that the white 'A' area is the iron matrix as the weight percentage of iron was as high as 95 wt%. Furthermore, the grey 'B' area contained approximately 40 wt% of the enriched Ti and C elements that comprise the TiC reinforcement. Therefore, good distribution of TiC reinforcement was achieved when milling was performed for 30-40 h.

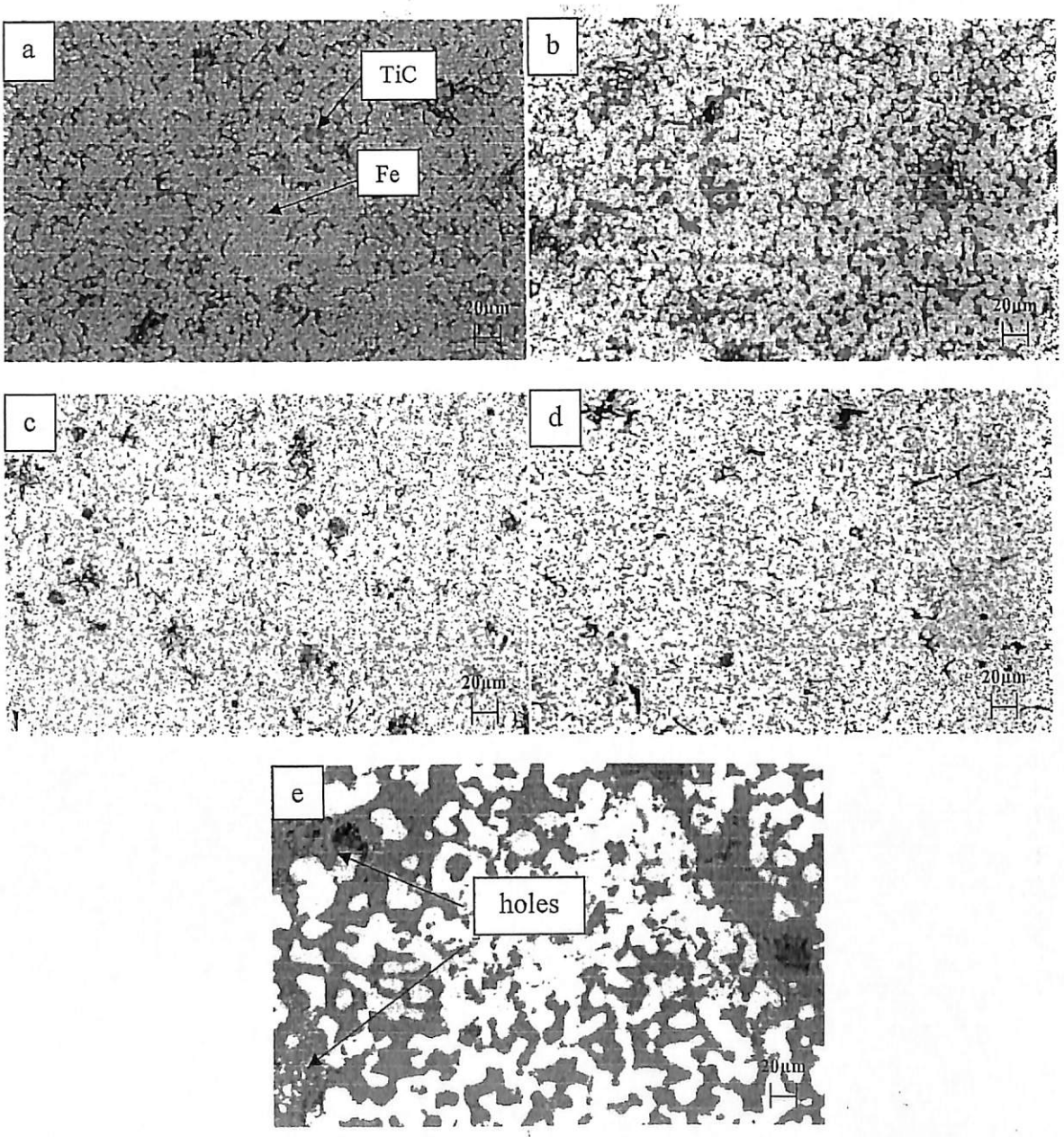
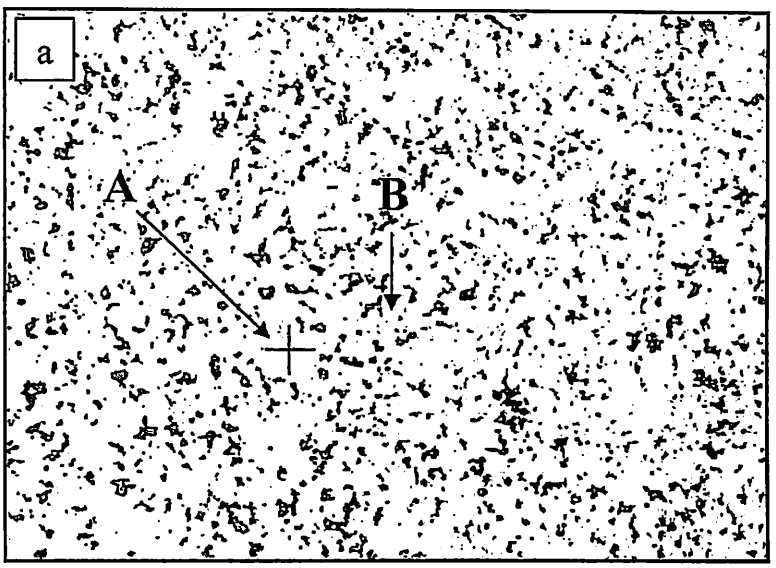
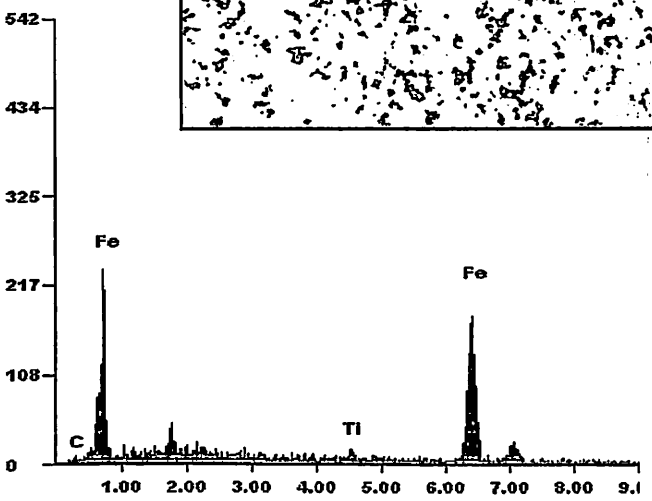


Fig. 7. Microstructure of sintered pellet of Fe-TiC composite with different milling times: (a) 10 h, (b) 20 h, (c) 30 h, (d) 40 h and (e) 60 h

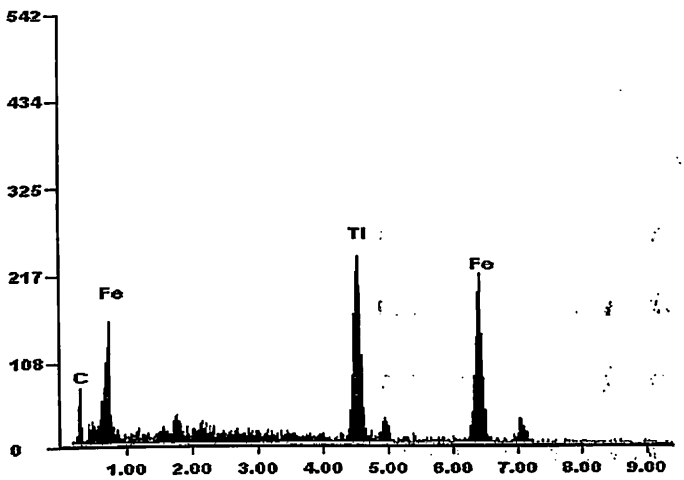


b



Element	Wt%	At%
Fe	94.78	86.38
Ti	02.67	02.84
C	02.54	10.78

c



Element	Wt%	At%
Fe	59.40	39.17
Ti	27.71	21.30
C	12.89	39.52

Fig. 8. (a) SEM image and corresponding energy dispersive X-ray (EDX) analysis of (b) 'A' area and (c) 'B' area of sintered Fe-TiC composite milled for 40 hours

The TiC reinforcement phase appears to have a good interface with the iron matrix [3], since the iron matrix coats the TiC phase very well without gap or porosity. Pagounis et al. [1] have reported that titanium carbide binds very well with the iron matrix, possibly because of the thermodynamic stability of titanium carbide in an iron matrix. The formation of cavities may have been caused by CO and CO_2 gases released during heat treatment, the source of porosity formation in the sintered pellet. The cavities or voids (black areas) in the iron matrix reduced in size with increasing milling time. However, prolonging milling until 60 h caused considerable morphological differences in the microstructure of the Fe-TiC composite, probably related to the change in the activated powder after milling from hematite to magnetite phase.

3.3 Particle size of as-milled powders

The change in particle size in the as-milled powder with milling time (Fig. 9) is explained by the stage of fracturing and rewelding of the mixture. Without milling, the particles were much coarser. The decrease in the particle size after 40 h milling was caused by successive ball to powder impacts, resulting in the progressive refinement of powder particles due to fracturing [18]. However, at 60 h milling, particle size increased and coarsened slightly compared to that of powders with 40 h milling due to agglomeration and rewelding of the highly refined powder during milling. The presence of magnetite phase after 60h milling could be the reason for an increase in particle size.

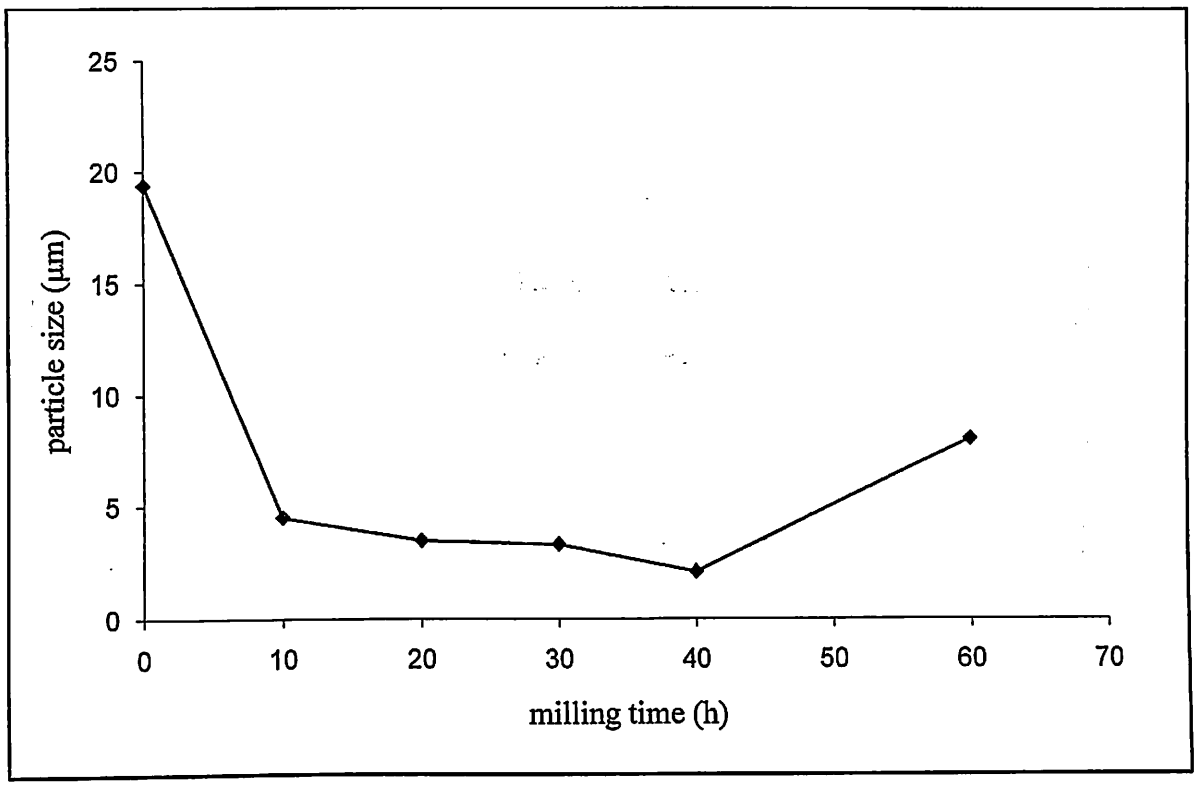


Fig. 9. Particle size of activated powder at different milling times

3.4 Density measurement

Fig. 10 shows the density measurements for consolidated and sintered pellets. As shown by the graph, the consolidated pellets had lower density than that of the sintered pellets. The lower density of consolidated pellets is caused by the presence of pores due to the low pressure (100 MPa) applied on the powder during consolidation. After sintering, the pellet exhibited higher density. The number of pores in the consolidated pellet decreased as the sintered pellet became denser due to atomic diffusion during high temperature sintering, which accelerated the particle-particle bonding formation. However, at longer milling time (60 h), the densities for both consolidated and sintered pellets suddenly dropped. Density in the 60 h milled consolidated pellets was much lower than that of other milled powders, thus lowering the density of the sintered composite.

This decrease in density resulted from the coarser particle of the activated powder at 60 h milling. The coarseness of the particles decreased the number of mechanical contacts formed among the particles during powder consolidation in forming the consolidated pellet [19]. The sudden drop in sintered density of the 60 h milled powder can be explained by the formation of open pores, as shown by SEM micrograph [Fig. 7(e)].In addition, the presence of magnetite phase instead of hematite phase in the 60 h milled powder also contributed to the drop in sintered density since the physical properties of magnetite and hematite are different, so the compressibility of magnetite- and hematite-containing powders and consequently their sinterability are also totally different.

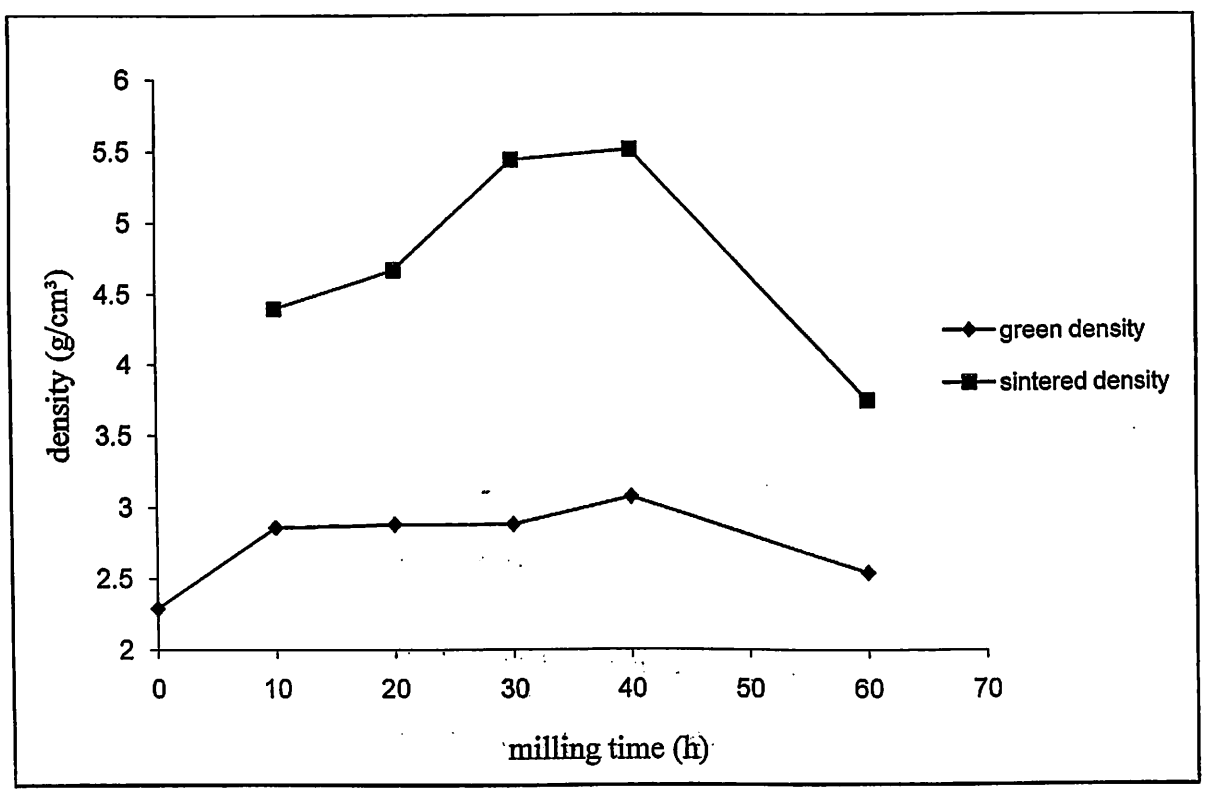


Fig. 10. Green density and sintered density of Fe-TiC compact at different milling times

3.5 Microhardness of Fe-TiC composite

Results of Vickers microhardness test of the Fe-TiC composite are shown in Fig.11. The trend seen in the results appears to be that the hardness of titanium carbide-reinforced iron composite that was developed via carbothermal reduction of hematite and anatase increased with increasing milling time. After sintering, the hardness of the non-milled sample could not be measured because sintering produced a porous iron matrix via reduction of hematite [19]. The increment of hardness in the milled composite is attributable to the formation of TiC reinforcement in the iron matrix as well as to the fineness of the powder. This agrees well with the findings of Ramesh et al. [20] since the hardness of the soft iron matrix can be improved by adding ceramic particle reinforcement. The homogenous distribution of fine TiC particle reinforcement in the iron matrix resulted in enhancement of the composite hardness. In addition, the increase in strain and dislocation with prolonged milling time significantly improved the hardness of the composite prepared with longer milling times. However, at60 h milling time, the hardness value decreased slightly, as can be explained by the transformation of hematite phase to magnetite phase as well as by the presence of pores in its microstructure.

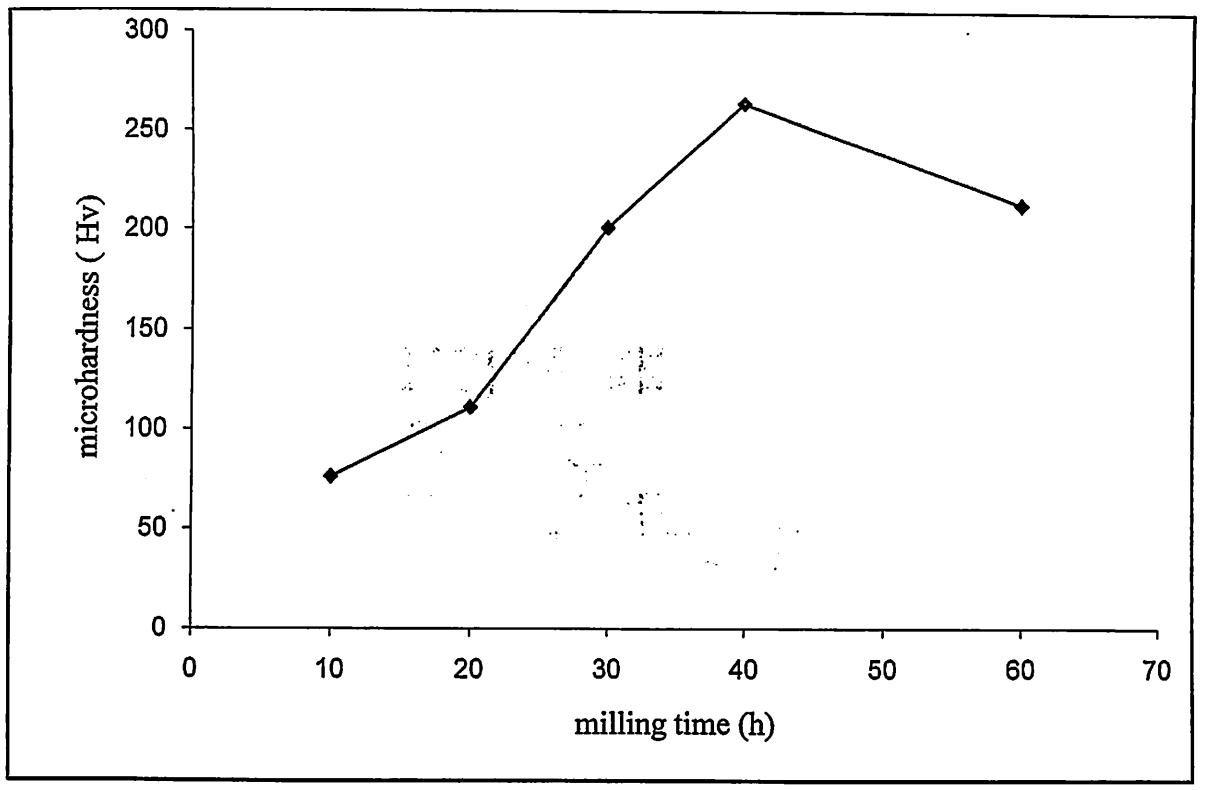


Fig.11. Microhardness (Hv) of Fe-TiC composite made with different milling times and sintered at 1100°C

Based on the findings, formation of Fe-TiC composite through carbothermal reduction of hematite and anatase mixture in this work can be proposed according to Fig. 12 which involves three stages, which are:

(a) Mechanical milling of Fe_2O_3-TiO_2 -C mixture for particle size reduction and increment of activation energy

(b) Reduction of Fe_2O_3 and TiO_2 by C and CO gas which has been formed through reaction between oxygen and graphite during sintering at 1100°C

(c) Formation of Fe-TiC composite since the formation of TiC is more favorable due to lower Gibbs energy compared to the formation of FeC.

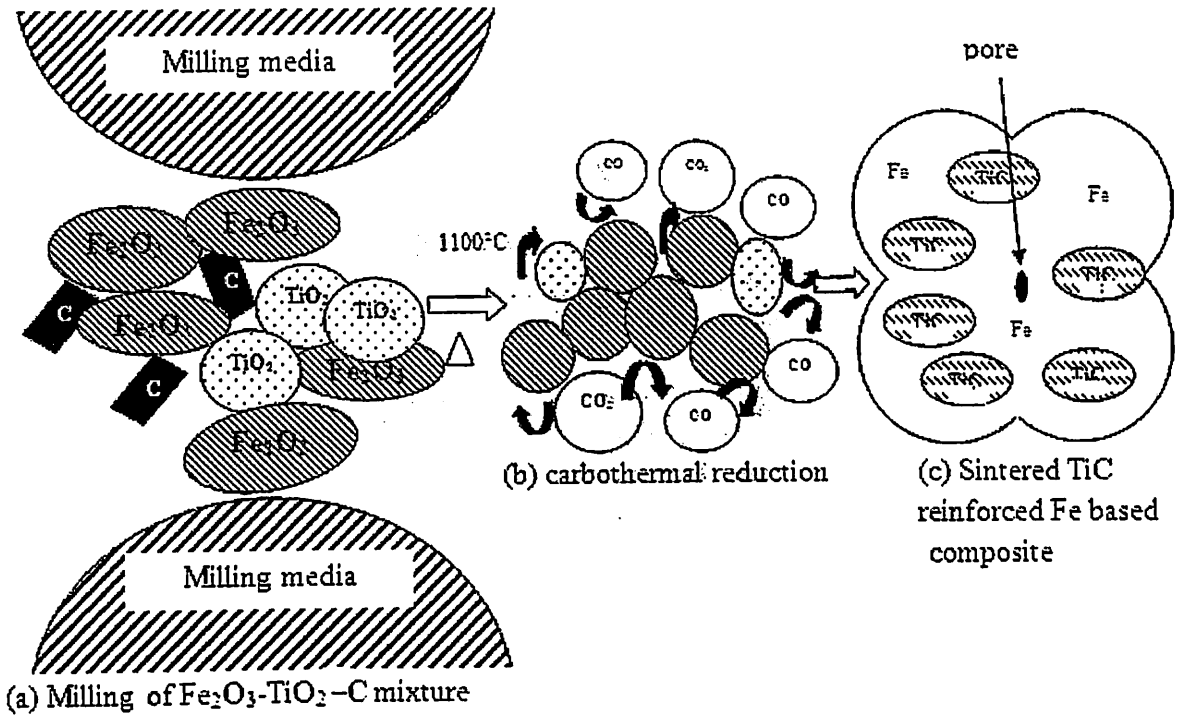


Fig.12. Schematic of Fe_2O_3-TiO_2 -C carbothermal reduction mechanism

4. Conclusion

Mechanical milling of hematite (Fe_2O_3) and anatase (TiO_2) mixture with graphite as reductant agent produced hematite (Fe_2O_3) phase in activated powder that had been refined, forming an amorphous structure after milling up to 40 h. After 60 h milling, magnetite (Fe_3O_4) peaks appeared in the XRD pattern as a result of phase transformation by reduction of hematite with graphite. As milling time increased, crystallite size, internal strain and average particle size decreased. The reaction forming TiC did not occur during sintering at 1100°C without mechanical milling, although the sintering temperature was higher than 900°C, the minimum temperature for the reaction to take place, which suggests the importance of mechanical milling in reducing the reaction temperature. SEM observation and EDX analysis of sintered Fe-TiC composite showed a homogenous distribution of TiC reinforcement throughout the iron matrix after 30-40 h of milling time. The Fe-TiC

composite developed using this method showed the good interfacial bonding that is required for good properties of the metal matrix composite. Density and hardness of the composite increased as milling time increased up to 40 h due to the formation of fine TiC and its homogenous distribution in the iron matrix. However, the formation of magnetite in the as-milled powder after 60 h milling reduced the density and hardness of the Fe-TiC composite.

5. Acknowledgement

The authors are pleased to acknowledge the financial support for this research by Universiti Sains Malaysia (USM) Fellowship for the first author as well as RU-PGRS research grant (8043042) and FRGS research grant (6071194).They gratefully acknowledge the helpful suggestions of Dr. Sheikh Abdul Rezan Sheikh Abdul Hamid of Universiti Sains Malaysia.

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UNIVERSITI SAINS MALAYSIA

LIST OF OUTPUT OF FUNDAMENTAL RESEARCH GRANT SCHEME (FRGS)

CARBOTHERMAL REDUCTION OF MECHANICAL ACTIVATED HEMATITE AND ANATASE MIXTURE FOR SYNTHESIS OF IRON BASED COMPOSITE

Project Number: 6071174

Duration: 1 MAY 2010 – 31 APRIL 2012

Project Leader: Dr. Zuhailawati Hussain

School of Materials and Mineral Resources Engineering,
Engineering Campus,
Universiti Sains Malaysia
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Penang
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Experimental Procedure

Hematite (Fe_2O_3), titanium oxide (TiO_2) and graphite (C) were used as raw materials. The mixture of raw materials which correspond to Fe-20vol%TiC were milled by using a high-energy Fritsch Pulverisette P-5 planetary mill in various milling time i.e. 0 h (non-milled), 20 h, 40 h and 60 h. The weight ratio of ball to powder was 10:1. The milling process was done in argon atmosphere. XRD analysis was carried out in order to identify phase formation in the as-milled powder. The as-milled powder was compacted under 100MPa of pressure by using cold press machine for consolidating the as-milled powder. The pellet was sintered in a vacuum furnace at 1100°C of sintering temperature for 1 hour and then cooled in furnace to room temperature. The sintered pellet was analyzed by X-Ray diffraction (XRD) analysis to investigae the formation of TiC in iron matrix after carbothermal reduction of oxides material. The microstructure of sample also was observed by scanning electron microscopy (SEM) analysis and the composition of sample was analyzed by energy dispersive X-ray (EDX) analysis.

Results and Discussion

XRD patterns of the sample containing Fe_2O_3, TiO_2 and C which was milled for 0-60 h are shown in Fig. 1. The results show that without milling (0 h), peaks of Fe_2O_3, TiO_2 and C are clearly detected. After milling (20 h-40 h), the intensity of all peaks reduced with increasing milling time. 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 20 h milling time is in agreement with the results of previous study [7]. All the peaks of as-milled powder were continuing decreased in intensity and increase in broadening by increasing milling time from 20 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_3O_4). This occurs because of the required surface free energy is enough to change the phase and modified structure of hematite to magnetite. The similar result also achieved by previous research whereby hematite phase changed to magnetite phase after 70 h milling in high energy milling [8].

Fig. 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 and C peaks were detected but TiC peaks were not detected. However after milling for 20 h and above, both Fe and TiC phases were identified. It is noted that the broadening of Fe and TiC peaks of 20 h until 40 h was slightly the same. This result suggests that without applying heat, the internal energy of the particles obtained during mechanical activation is not enough to initiate the reaction to form Fe and TiC phases. At 0 h, phase of Ti_2O_3 was detected because effective mechanical activated powder particles cannot be achieved by conventional mixing process. Through high energy ball milling, the energy transferred between powder particles and milling media was more efficient. It was reported by Schaffer and Forrester [9] that the collision frequency plays a role to produce effective collision event. This produces the activated powders which can easily react with carbon as mechanically activated carbon become an effective reductant for Fe_2O_3 and TiO_2 [1].

Fig. 3(a) shows microstructure observation of Fe-TiC composites after sintering at 1100°C. The sample milled for 40 hours shows the distribution of iron and TiC enriched areas. The white region ('A' area) representing iron matrix and the dark region ('B' area) represent rich side of titanium carbide. This conclusion is supported by energy dispersive X-ray (EDX) analysis where Fig. 3(b) shows the higher percentage of iron matrix composition in white region whilst Fig. 3(c) represent for dark area shows the enrichment of titanium carbide reinforcement formation. It is observed that

a homogeneous distribution of titanium carbide reinforced iron based composites was achieved at 40 hours milling time and sintered at 1100°C.

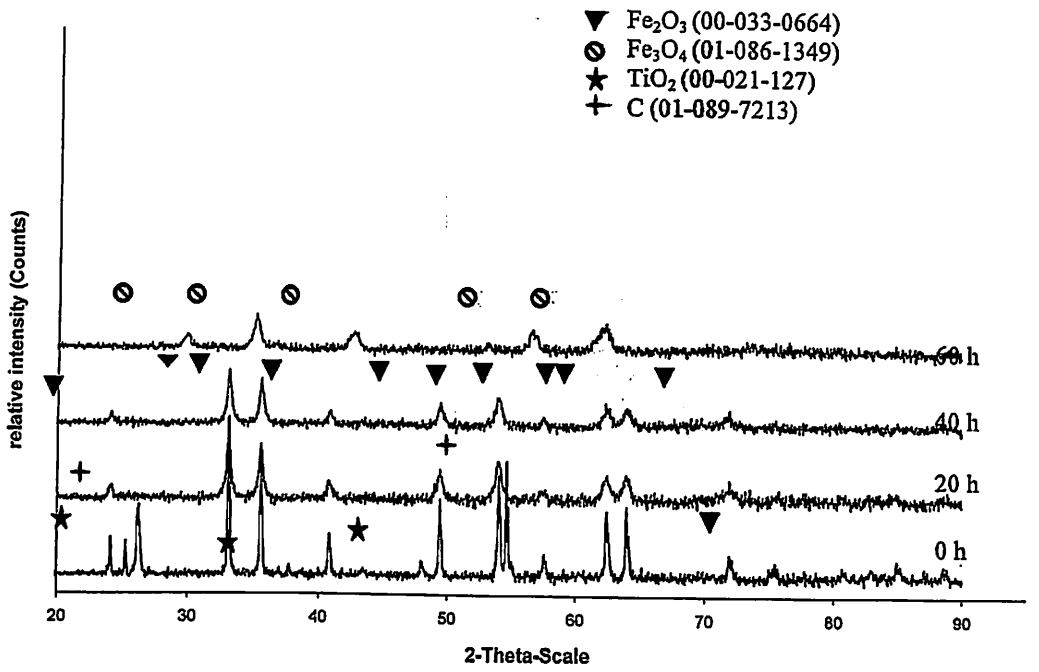


Fig. 1: XRD patterns of Fe_2O_3-TiO_2 -C powder as-milled with milling times of 0 h, 20 h, 40 h and 60 h

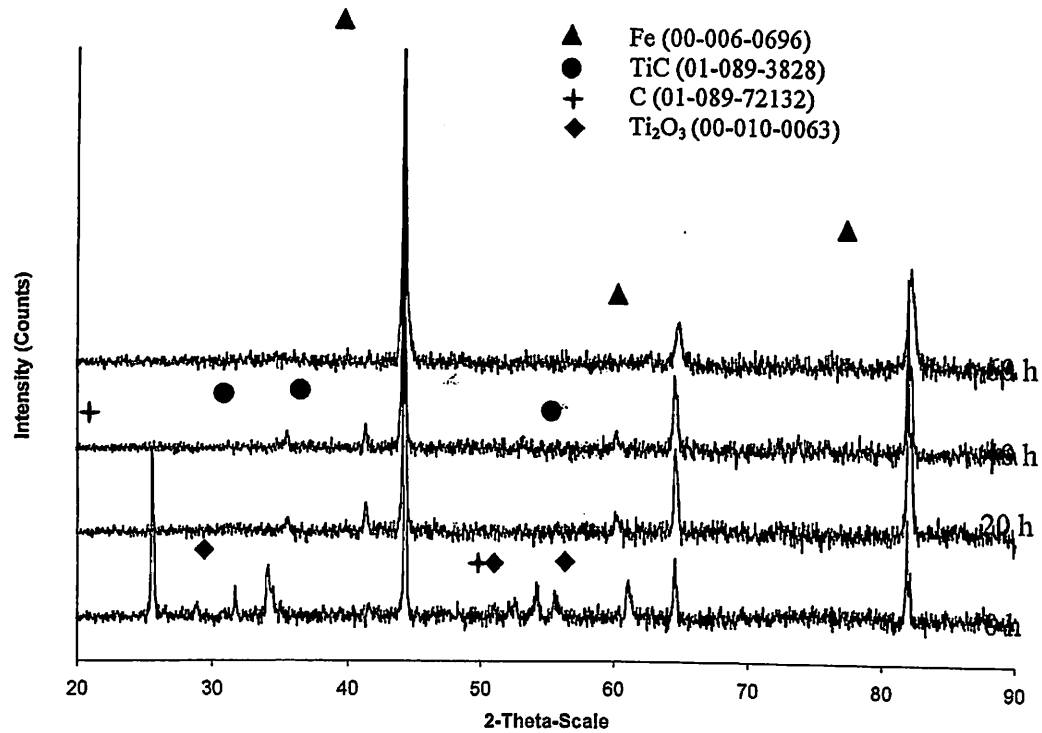
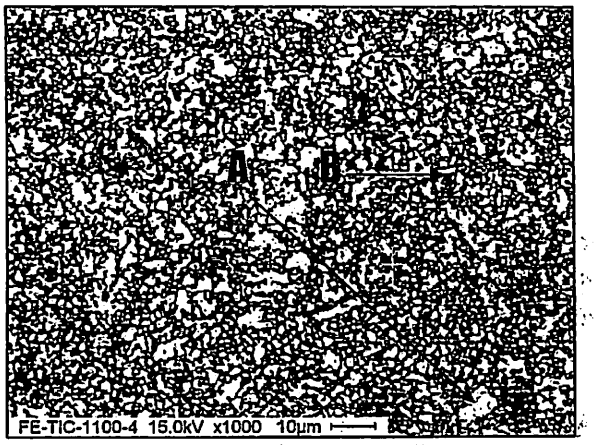
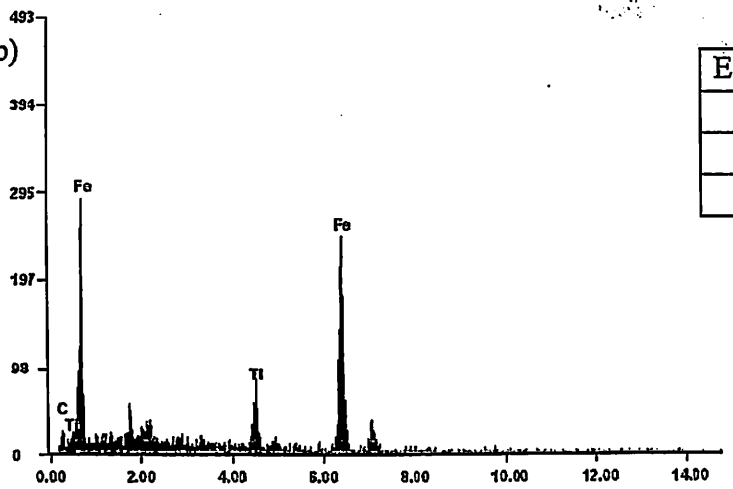


Fig. 2: XRD patterns of sintered Fe-TiC with milling times of 0 h, 20 h, 40 h and 60 h

(a)

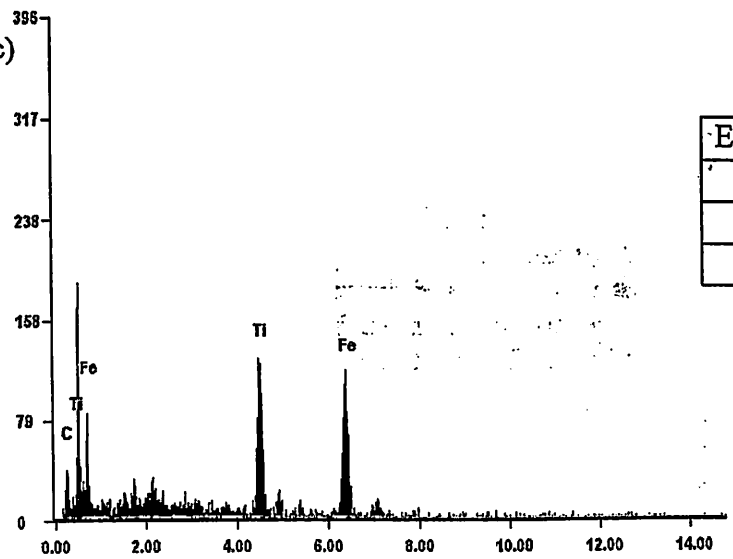


(b)



Element	Wt%	At%
C	06.42	23.88
Ti	09.71	09.06
Fe	83.87	67.07

(c)



Element	Wt%	At%
C	15.56	44.92
Ti	25.57	18.51
Fe	58.88	36.56

Fig. 3: (a) SEM image and corresponding energy dispersive X-ray (EDX) analysis at (b) 'A' area and (c) 'B' area of sintered Fe-TiC composite milled for 40 hours of milling time

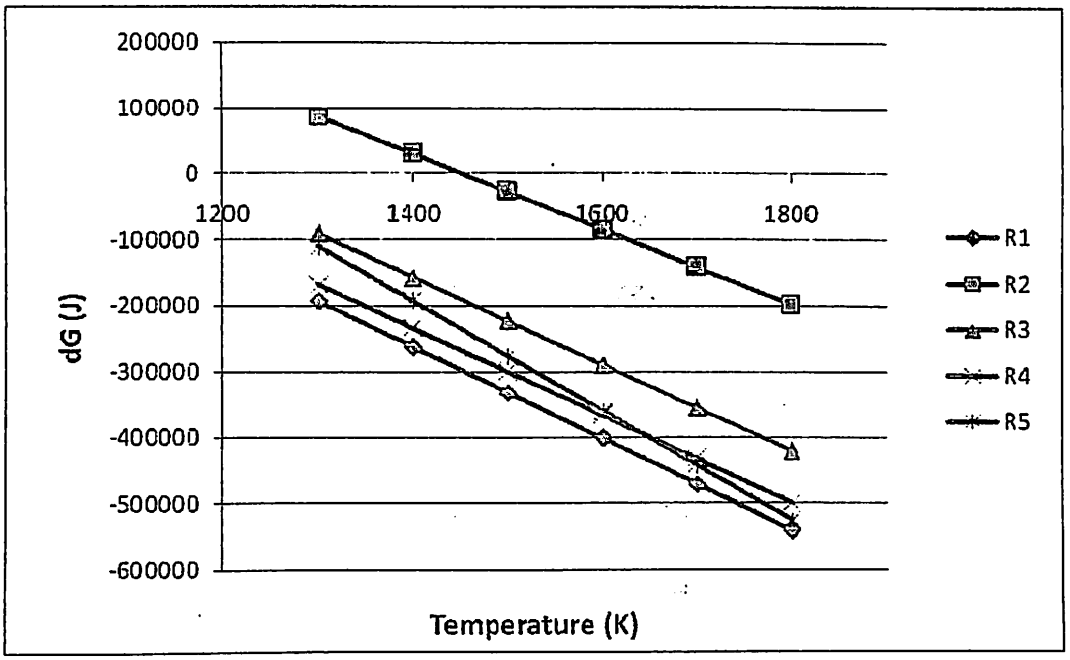


Fig.4: Gibbs energy (ΔG) vs temperature of formation Fe-TiC from hematite and anatase

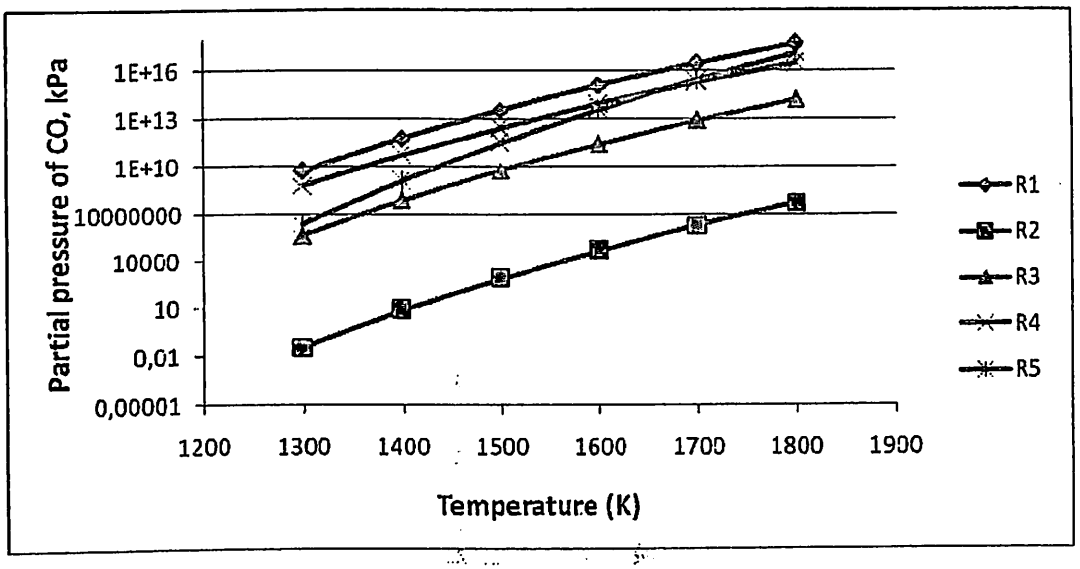


Fig.5: Partial pressure of CO vs temperature of formation Fe-TiC from hematite and anatase

R1: $Fe_2O_3 + 3TiO_2 + 4C \rightarrow 2Fe + Ti_3O_5 + 4CO$
R2: $Fe_2O_3 + 2\ Ti_3O_5 + 3C \rightarrow 2Fe + 3Ti_2O_3 + 3CO$
R3: $Fe_2O_3 + Ti2O3 + 4C \rightarrow 2Fe + 2TiO + 4CO$
R4: $Fe_2O_3 + Ti_2O_3 + 5C \rightarrow 2Fe + TiC + 4CO$
R5: $Fe_2O_3 + TiO_2 + 6C \rightarrow 2Fe + TiC + 5CO$

Fig.4 shows Ellingham Diagrams of formation Fe-TiC from hematite and anatase at 1100 °C (1373 K) based on thermodynamics calculation theory. Based on R5, minimum temperature of reduction reaction is 894.15 °C to form Fe-TiC and Gibbs energy (ΔG) for overall reaction of carbothermal reduction is about -19×10^6 J. Therefore, it is possible to form Fe-TiC composite at 1100 °C which has been proved by XRD analysis. Fig.5 shows partial pressure of CO vs temperature of formation Fe-TiC whereby the pressure released by CO for R5 is below than 13 x

10^6 kPa which indicates that the reaction goes forward as predicted by Le Chatelier's principle [10-11].

Summary

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 (Fe_3O_4) peaks appeared in XRD pattern sample milled for 60 h which indicated that some of hematite particles have been reduced to magnetite due to milling in the presence of graphite. Mechanical activated sintering at 1100 °C of the Fe_2O_3- TiO_2-C compact transformed the phases to Fe-TiC composite with enhanced intensity of Fe peaks in XRD patterns. The formation of Fe-TiC composite was also confirmed by SEM examination and EDX analysis.

Acknowledgement

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X-ray and Related Techniques
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Formation of TiC-Reinforced Iron Based Composite through Carbothermal Reduction of Hematite and Anatase
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Sintering of Fe-TiC Composite Prepared by Carbothermal Reduction of Hematite and Anatase

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Keywords: Hematite; Fe-TiC composite; Carbothermal reduction; Sintering temperature

Abstract. In this study, the formation of Fe-TiC composite through carbothermal reduction of hematite and anatase was investigated with various sintering temperature. Mixture of hematite and anatase powders was milled with graphite for 20 hours in a high energy ball mill in argon atmosphere with composition of Fe-30%volTiC. The as-milled powder was analyzed with X-ray diffraction analysis and the result shows broadening of hematite peaks with disappearance of anatase and graphite peaks due to refinement of powder and diffusion of carbon. The as-milled powder was cold pressed under 200 MPa and sintered in argon atmosphere at various sintering temperature i.e, 1200°C, 1300°C and 1400°C. Higher sintering temperature facilitated reduction of hematite and anatase to produce Fe-TiC composite.

Introduction

Development of iron based composite considerably attracts many interests due interest due to an outstanding properties for wear resistance in chemical and process industries application [1,2]. Iron based composite commonly reinforced by ceramic materials such as Al_2O_3, ZrO_2, TiN_2, Si_3N_4 and TiC [1]. Among ceramics reinforcement, TiC has proved its suitability in Fe. TiC is extensively used in iron matrix because its extremely good properties such as good wettability, high hardness, low density and thermal stability since it is useful for manufacturing of cutting tool, grinding wheels and steel-coated press tools [1,3,4].

There are several routes for fabrication of iron based composites such as powder metallurgy, conventional melting and casting and carbothermal reduction route [1]. The carbothermal reduction of iron oxides by reductant materials performed by mechanical activation improved the rate of reduction because energy during mechanical milling provides a driving force for reaction to take place [5,6]. The energy transferred to activated powder during milling influences kinetic of chemical reactions between powder due to an increase in crystal defect such as dislocation density and structural distortion which are associated with the formation of amorphous structure [5]. For example, synthesis of Fe-TiC by reduction of ilminite (FeTiO3) with graphite successfully occurred at 1250°C after 50 h milling [6]. Without milling, phase of Fe and TiC cannot be observed by XRD analysis after 1250°C of heat treatment temperature [6]. Meanwhile, TiC is commercially produced by reduction of TiO_2 by carbon in a temperature range between 1700°C and 2100°C [7].

Besides the reduction of ilmenite (FeTiO3), the possible carbothermal reduction of hematite and anatase with graphite result in formation of Fe-TiC composite should occurs as summarized in Eq. 1:

$$Fe_2O_3 + TiO_2 + 6C \rightarrow 2Fe + TiC + 5\,CO \quad (1)$$

Thus, the aim of this study is to investigate the suitable heat treatment temperature of formation iron based composite reinforced titanium carbide by using iron oxide (Fe_2O_3), titanium dioxide (TiO_2) and graphite. The temperatures selected are in the range for solid state sintering.

Experimental Procedure

A mixture of hematite (Fe_2O_3), anatase (TiO_2) and graphite were mechanically milled together in argon atmosphere by using a high-energy Fritsch Pulverisette P-5 planetary mill for 20 hours milling time. The milling speed was set at 400 rpm. The weight ratio of ball to powder was 10:1. The activated milled powder which corresponds to Fe-30%TiC was analyzed with X-ray diffraction (XRD) analysis in order to identify phase presence in the as-milled powder. The milled powder was consolidated under 200 MPa of pressure by using cold pressing machine. The pellets was heat treated in tube furnace with argon flow in various temperature i.e 1200°C, 1300°C and 1400°C at heating rate of 10°C/min for 1 hour of soaking time and cooled in furnace to room temperature. X-Ray diffraction (XRD) analysis was done on the sintered pellets to investigate the formation of TiC in iron matrix after carbothermal reduction of oxide metals at high temperature.

Results and Discussion

Fig. 1 presents the XRD pattern of the as-milled powder after 20 hours milling time. The phase detected by XRD in the sample is only hematite (Fe_2O_3). The disappearance of TiO_2 and C phases in the as-milled powder most probably because of the formation of amorphous structure during mechanical milling since the powder particle undergo repeated flattening, cold welding and fracturing [8]. The absent peaks of C and TiO_2 also indicate that these phases are easily deformed and changed into an amorphous structure compared with the harder Fe_2O_3 phase after milling [4]. The mixture cannot be reduced to form Fe and TiC phases after milling because Fe_2O_3 and TiO_2 are very stable phases and formation of Fe-TiC composite needs a larger amount of energy that cannot be provided by milling process.

X-ray diffraction of sintered pellet of difference heat treatment temperature is shown in Fig. 2. The presence of Fe and TiC indicate the successful of carbothermal reduction of Fe_2O_3 and TiO_2 after sintering. The intensity of iron and titanium carbide peaks increased with increasing sintering temperature. At 1200°C of heat treatment, the intensity of Fe and TiC peaks is low compared with higher temperature. This situation occurred because of a significant effect in enhancement of reduction rate with increasing temperature. Increasing the carbothermal reduction temperature to 1400°C significantly enhanced the formation of TiC phases. Stronger intensity of Fe and TiC peaks are obviously seen after sintering at 1400°C.

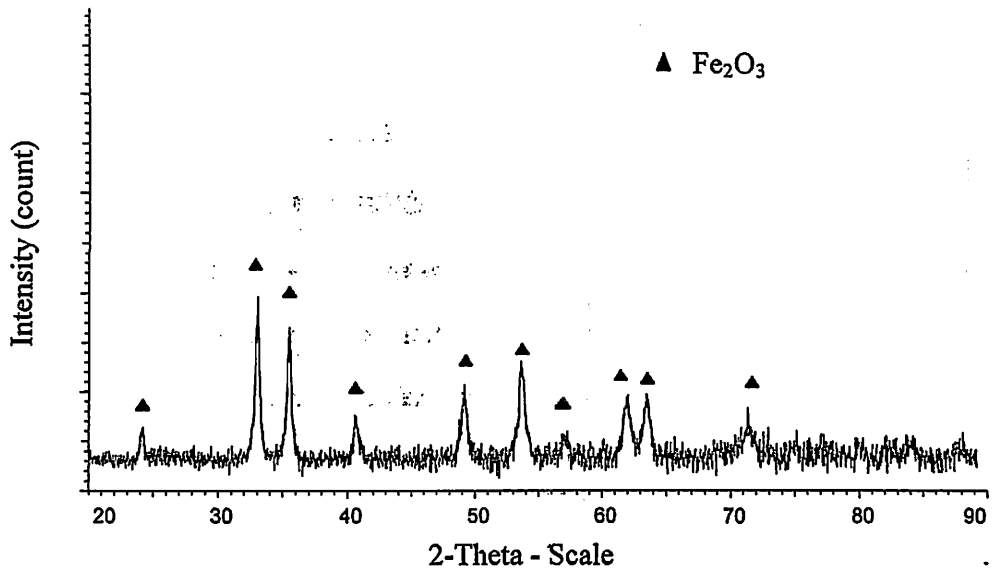

Fig. 1: XRD pattern of Fe_2O_3-TiO_2-C as-milled powder after 20 h milling

Mechanical milling of mixture plays the major effect of carbothermal reduction at lower temperature. The statement is well agree with Razavi and Rahimipour [6], who observed after 50 hours of mechanical milling, $FeTiO_3$ has been reduced by C to form Fe-TiC after sintering at 1250 °C compared with non-milled sintered pellet whereby the Fe-TiC was not observed at the same sintering temperature. Besides, mechanical milling also plays a function as a medium to overcome the solid-state diffusion limitation of the mixture by increasing the crystalline defect such as dislocation, small particle size, formation of amorphous phase and atom displacement which reduce the heat treatment temperature to form Fe-TiC composite [4,5].

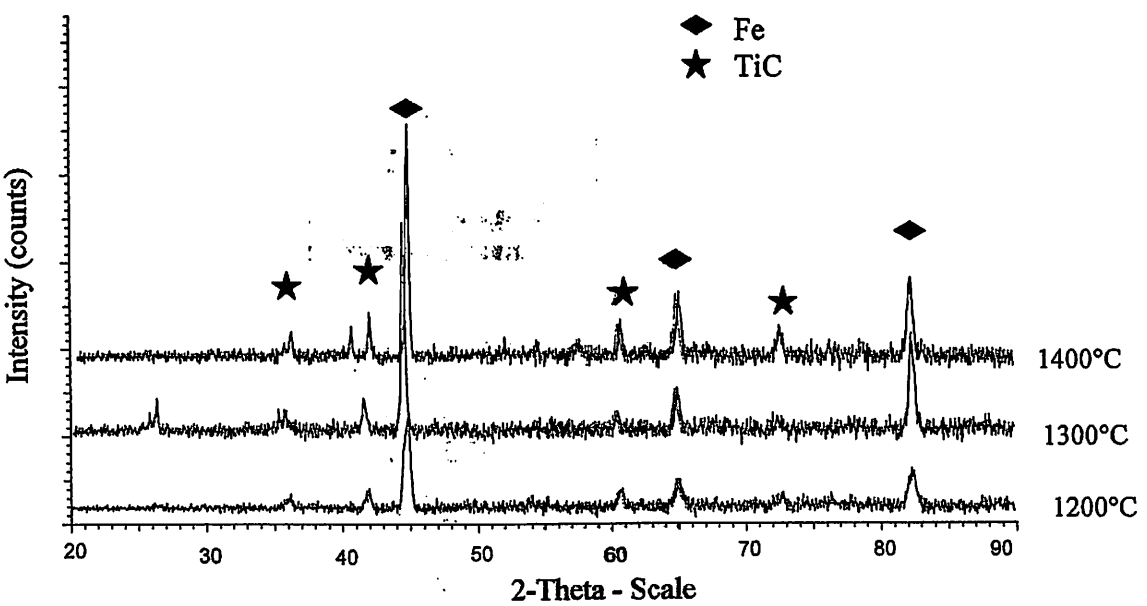

Fig. 2: XRD patterns of Fe-TiC composite formation at various heat treatment temperatures

Summary

In this study, the amorphous structure of hematite and anatase has been obtained after 20 hours of mechanical milling since TiO_2 and C phase not been. The carbothermal reduction of hematite and anatase was significantly affected by sintering temperature. The reduction rate increased with increasing sintering temperature. The formation of TiC-reinforced iron based composite of milled powder in argon flow was possible to take place at low temperature (1200°C) as shown in XRD pattern.

Acknowledgement

The authors are pleased to acknowledge the financial support for this research by Universiti Sains Malaysia (USM) Fellowship, RU-PGRS (8043042) research grant and FRGS (6071194) research grant.

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FUNDAMENTAL RESEARCH GRANT SCHEME
FINAL REPORT ASSESMENT FORM
Borang Penilaian Laporan Akhir
Skim Geran Penyelidikan Fundamental

A.	**TITLE OF RESEARCH:** *Tajuk penyelidikan:*	*Carbonthermal Reduction of Mechanical Activated Hematite and Anatese Mixture for Synthesis of iron Based Composite*
B.	**PERSONAL PARTICULARS OF RESEARCHER** / *Maklumat Penyelidik:*	
	(i) **Name of Research Leader:** *Nama Ketua Penyelidik:*	*Dr. Zuhailawati Hussain*
	(ii) **School/Institute/Centre/Unit:** *Pusat Pengajian /Institut/Pusat/Unit:*	*Pusat Pengajian Kejuruteraan Bahan & Sumber Mineral*
	(iii) **Phone No. (Office):** *No. Tel. (Pejabat):*	
	(iv) **Hand Phone No:** *No. Telefon Bimbit:*	
	(v) **Fax. No:** *No. Faks:*	

C.	**SUMMARY OF ASSESSMENT** *(Tick (√) the appropriate box. Also, provide additional comments in Section F)*	Inadequate		Acceptable	Very Good	
		1	2	3	4	5
1.	**Achievement of Project Objectives**	☐	☐	☑	☐	☐
2.	**Quality of Output**	☐	☐	☐	☑	☐
3.	**Quality of Organisational outcomes**	☐	☐	☑	☐	☐
4.	**Quality of sectoral/national impacts**	☐	☐	☑	☐	☐
5.	**Technology Transfer / Commercialization Potential**	☐	☐	☐	☐	☐
6.	**Quality and Intensity of Collaboration**	☐	☐	☐	☐	☐
7.	**Overall Financial Expenditure**	☐	☐	☐	☑	☐
8.	**Overall Assessment of Benefits**	☐	☐	☑	☐	☐

D.	OUTPUT SUMMARY				
1	Achievement Percentage				
	Project progress according to milestones achieved up to this period	25%	50%	75%	100%
					✓
2	Research Findings				
		ISI Journal	International Journal	National Journal	
	Number of articles/ manuscripts/ books	2			
		International	National		
	Paper presentations	2			
	Others (Please specify)				
3	Human Capital Development				
		Number			
		On-going		Graduated	
		M'sian	Non- M'sian	M'sian	Non- M'sian
	Ph D Student				
	M Sc Student	2			
	Undergraduate Final Year Project	1			
	Temporary Research Officer				
	Temporary Research Assistant				
	Others (Please specify)				
	Total				

E.	ACTION TO BE TAKEN *(Tick (√) the appropriate boxes)*	
	☑	**Project file to be closed.** *(For financial and administration purposes)*
	☐	**Project to be Reviewed Once Additional Information Has Been Obtained by the Project Leader** *(Please see section E)*
	☐	**Forward to Innovation Office for Consideration:** ☐ **Patent** ☐ **Commercialisation** ☐ **Technology Transfer** ☐ **Others** *(Please specify):* _____
	☐	**Forward to Division of Industry & Community Network (DICN)**
	☐	**Others** *(Please specify):* _____ *(E.g.: Formations of teams or cluster etc.)*

F. **Additional information to be provided by the Project Leader** [Please provide details of the specific information being requested from the project Leader in the areas identified in Section E]

G. **Comments Regarding Assessment** [Please provide below an explanation of any assessment made in Section C showing a rating below "acceptable"]

H. **Overall Comments:**

- Provide copies of full papers published under this grant.
- Provide copies of their abstracted for graduate students.

Name of Panel: ~~PA Research H Head~~ **Signature** : _____

Date : 9/4/2012

I. **Endorsement & Comments/Suggestions of Research Dean:**

Signature: _____ **Date** : _____