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Fabrication of *in situ* Fe-NbC Composite by Mechanical Alloying

(Fabrikasi Komposit Fe-NbC *in situ* Secara Pengaloian Mekanikal)

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ABSTRACT

In this research we investigated the in situ *formation mechanism of NbC in mechanically alloyed Fe-Nb-C mixture. Powders of iron, niobium and graphite with a composition Fe-20 %Nb was milled in a planetary mill for various milling times (i.e. 5, 10, 15 and 20 h) to investigate the influence of this variable on phase formation and properties of composite. The mixture was cold pressed and sintered at 1300°C for 1 h. Only phase of the initial raw materials was observed after milling, whilst NbC phase was detected after sintering. Increasing the milling time resulted in an increase in crystallite size and strain energy, which is beneficial for hardness and density improvement.*

Keywords: Fe matrix; mechanical alloying; milling speed; NbC phase; powder metallurgy

ABSTRAK

Dalam penyelidikan ini, kami menerangkan mekanisme pembentukan NbC secara in situ *di dalam campuran Fe-Nb-C yang dialoi secara mekanikal. Serbuk besi, niobium dan grafit dengan komposisi Fe-20 %NbC (pecahan isi padu) dikisar di dalam pengisar planet untuk pelbagai tempoh (5, 10, 15 dan 20 jam) bagi menjelaskan pengaruh pemboleh ubah tersebut terhadap pembentukan fasa dan sifat komposit. Campuran ditekan sejuk dan disinter pada 1300°C selama 1 jam. Hanya bahan mentah awal sahaja yang diperhatikan selepas pengisaran manakala fasa NbC dikenal pasti terbentuk selepas pensinteran. Penambahan tempoh pengisaran menghasilkan pertambahan dalam saiz hablur dan tenaga terikan yang memberikan kekuatan dan memperbaiki ketumpatan.*

Kata kunci: Fasa NbC; halaju pengisaran; matriks Fe; metalurgi serbuk; pengaloian mekanikal

INTRODUCTION

In the metal matrix composite field, most previous investigations dealt with the non-ferrous matrix and only a few investigations focused on iron-based composite; particularly those that were developed *in situ* by mechanical alloying. Iron-based composite is suitable for automotive and manufacturing industries, such as high-speed cutting tools or grinding media (Zapata et al. 1995). Interfacial compatibility and serious interfacial reaction are the crucial issues that receive the most attention in research works on metal matrix composite, which can be overcome by an *in situ* approach during the incorporation of reinforcement particles in the metal matrix, with the help of mechanical alloying (Yilmaz et al. 2009).

A longer milling time is usually applied to encourage reinforcement formation. However, a combination of different milling parameters, such as milling speed, ball to powder weight ratio and other parameters can influence the milling time (Suryanarayana 2004). With prolonged milling, particle size increases when particles start to agglomerate and cold weld. Since only a few investigations have addressed iron based composite, more detailed information is required on the effects of milling time on iron matrix composite. Therefore, the aim of this work was to investigate the effect of milling time on the formation of *in situ* Fe-NbC composite and its properties.

EXPERIMENTAL DETAILS

A mixture of Fe-17.21 wt. %Nb-2.23 wt. %C; corresponding to Cu-20 vol% NbC, was milled using a Fritsch Pulverisette 5 planetary mill at 100 rpm and milling times of 5, 10, 15 and 20 h. Stainless steel balls (20 mm in diameter) were used, with a ball to powder weight ratio of 10:1. The as-milled powder was characterized using X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). The as-milled powder was compacted in a 10 mm diameter cylindrical stainless steel die, at a pressure of 600 MPa. The resulting pellet was sintered at 1300ºC in an argon atmosphere and characterized by XRD and SEM. A hardness test was carried out using a Future Tech Vickers Hardness Tester under a 1000 g load, with a dwell time of 10 s, whilst Archimedes' principle was used to measure the sintered density.

RESULTS AND DISCUSSION

PHASE ANALYSIS

Figure 1(a) shows the XRD pattern after mechanical alloying. Apparently, the starting materials of Fe and Nb were still observed in the XRD pattern after they had been milled for up to 20 h. XRD peaks of graphite were absent due to amorphization during mechanical alloying (Lou et al. 1996). Peak broadening was observed with increased milling time, indicating that powder refinement was a result of repeated ball collision on the powder particles (Ismail et al. 2011). The NbC phase was clearly absent, even though milling was conducted for up to 20 h; suggesting that activation energy for the reaction was not yet achieved.

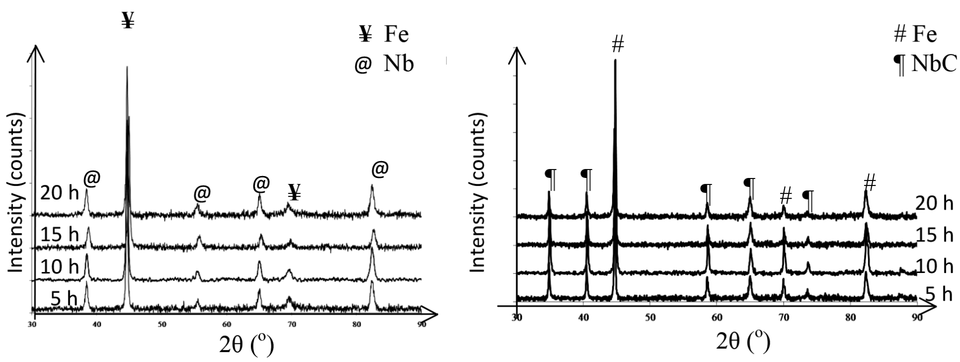
Figure 1(b) shows the XRD pattern of mechanical alloyed Fe-Nb-C powder, sintered at 1300°C. Heat energy during sintering overcome the activation energy to form the NbC phase. Longer milling time broadened the XRD peaks, without peak shifting and the appearance of new diffraction peaks. Neither the oxide or the contamination phase was present, due to the low energy of the milling speed that was used (Suryanarayana 2004).

MICROSTRUCTURE INVESTIGATION

Figure 2 shows the SEM micrograph of a sintered pellet under a back scattered electron mode where NbC phase indicated in grey. Prolonged milling caused the diffusion of Nb and C atoms, to form a fine-layer of NbC within the iron matrix, which improved homogeneity. EDX quantitative analysis suggests that diffusion of Nb and C occurred as a result of mechanical alloying to form fine- layer of NbC within the iron matrix.

COMPOSITE PROPERTIES

Figure 3 shows the crystallite size and internal strain of the Fe phase, calculated based on the XRD analysis.



(a) (b)

FIGURE 1. XRD patterns of Fe-NbC (a) as-milled powder (b) sintered pellet at different milling times of 5, 10, 15 and 20 h

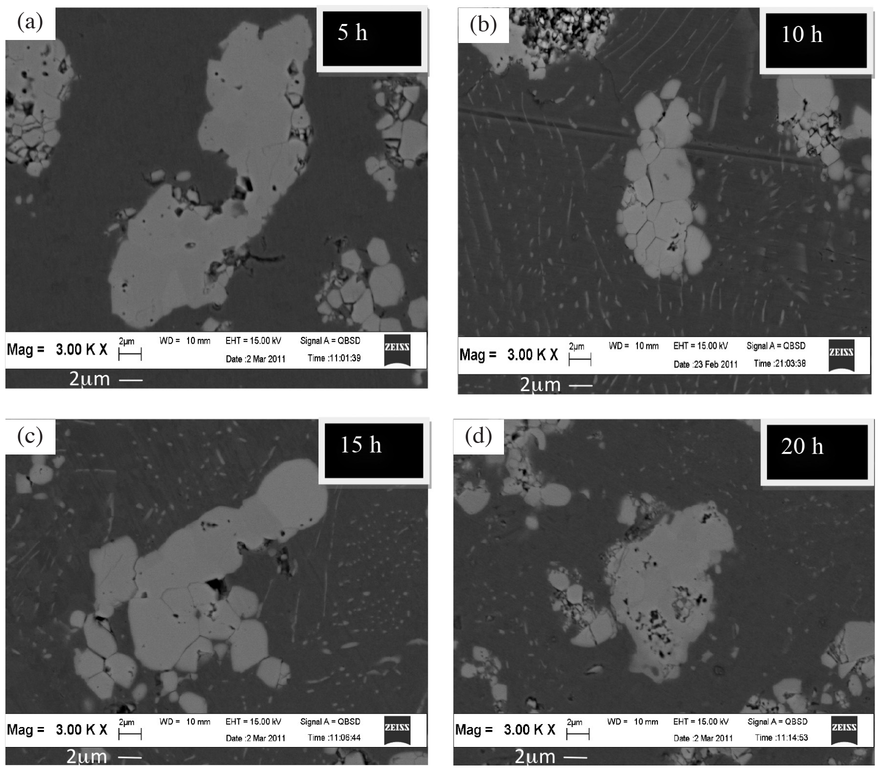


FIGURE 2. SEM images of sintered composite for (a) 5 h, (b) 10 h, (c) 15 h and (d) 20 h and EDX analysis on different points

Increasing milling time reduced the crystallite size and increased the internal strain. A longer milling time resulted in continuous impact on the powders and provided a repeated accumulation of dislocation, which then refined the crystallite size (Zuhailawati & Mahani 2009). The strain value increase was due to the generation of dislocation and other crystal defects (Suryanarayana 2004). However, the change was insignificant, which showed that milling at 100 rpm provided a low energy impact, even for longer times. Figure 4 shows the density measurement of sintered composite obtained using the Archimedes method. By increasing milling time, various crystal defects, such as dislocation, vacancies, stacking faults and grain boundary, increased; which enhanced the diffusivity of atoms during sintering.

Figure 5 shows that the microhardness of Fe-NbC composite increases with milling time, due to NbC dispersion in the Fe matrix, which prevented movement of dislocation in the iron matrix (Da Costa et al. 2003). Another reason for the increment of hardness value was the refinement of the NbC reinforcement. Prolonging the milling process also produced fine grains with a larger surface area of grain boundary (Hemanth 2009).

CONCLUSION

An investigation was made to study the effect of milling time on the formation and properties of *in situ* Fe-NbC composite, synthesized by mechanical alloying. A longer milling time does not contribute to the formation of NbC

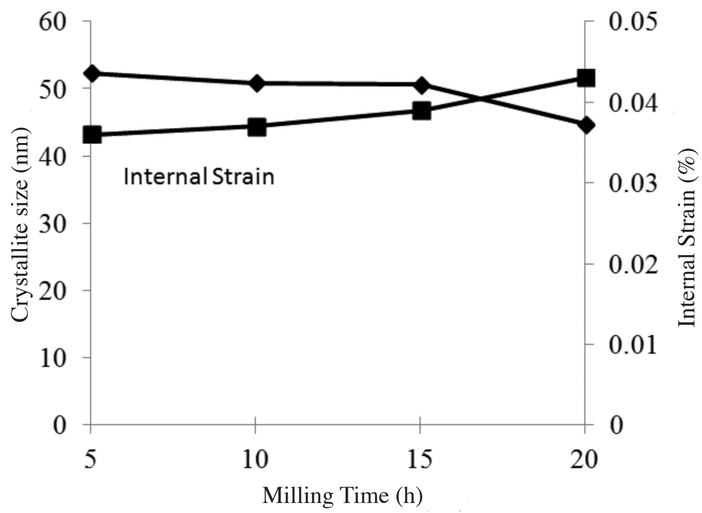


FIGURE 3. Crystallite size and internal strain of Fe–NbC sintered composite at different milling times

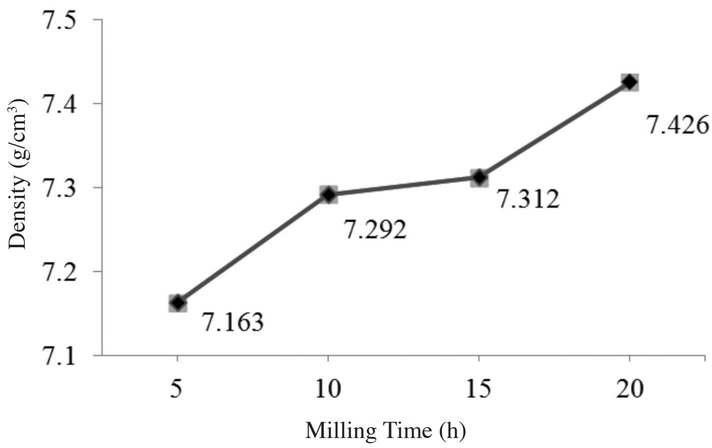


FIGURE 4. Density measurement of Fe–NbC sintered pellet at different milling times

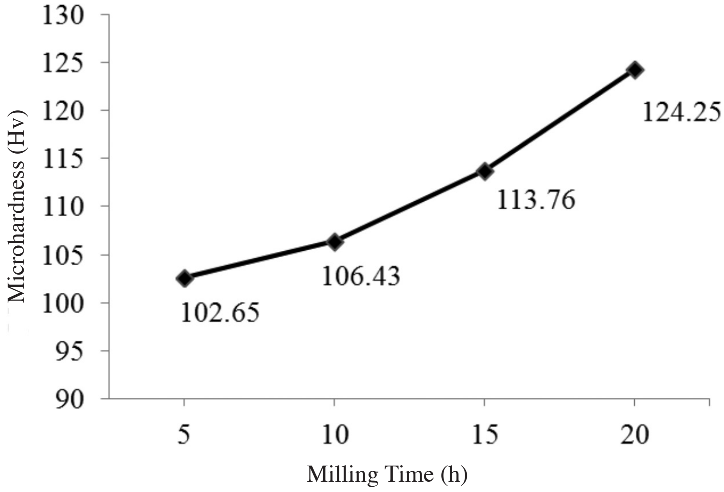


FIGURE 5. Microhardness measurement of Fe-NbC sintered pellet at different milling times

phase after milling. After sintering at 1300°C, increasing the milling time caused the NbC phase to become finer, which is good for density and hardness improvement.

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