

## Novel Aluminium Based Composite Materials Exhibiting Magnetic Properties Fabricated by Mechanical Alloying and Spark Plasma Sintering

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Functionally aluminium based composite materials with exhibiting magnetic properties were fabricated using a mixture of pure aluminium and ferrite powder via mechanical alloying (MA) and subsequently spark plasma sintering (SPS). Changes in hardness, constituent phases and soft magnetic properties of both mechanically alloyed powders and spark plasma sintered materials were examined by microhardness measurements, X-ray diffraction and vibrating sample magnetometer, respectively. Effects of compositions, MA time and sintering temperatures on the properties of the SPSed materials were investigated. The Vickers microhardness of MAed powders increased by an increase of both MA time and the amount of ferrite powders. No solid-state reaction for all MAed powders was detected during MA process. The magnetic induction clearly exhibited to depend upon the amount of ferrite powder.

**Keywords:** *mechanical alloying, magnetic properties, aluminium, composite material, spark plasma sintering.*

### 1. Introduction

Aluminium and its alloys that, are negligibly affected by magnetic field, are known non-magnetic substances. Exhibiting magnetic properties of aluminium alloys can be used for a variety of applications, such as automobile industries. Ferrites are most widely used as magnetic materials that are ceramic with  $\text{Fe}_2\text{O}_3$  as their principal components. Among them, many ferrites are spinels with the formula  $\text{AB}_2\text{O}_4$ , where A and B represent various metal cations, usually including iron. Spinel ferrites usually adopt a crystal motif consisting of cubic close-packed oxides with A cations occupying one eighth of the tetrahedral holes and B cations occupying half of the octahedral holes. That is, the inverse spinel structure.

Ferrites are usually non-conductive ferrimagnetic ceramic compounds derived from iron oxides such as hematite ( $\text{Fe}_2\text{O}_3$ ) or magnetite ( $\text{Fe}_3\text{O}_4$ ) as well as oxides of other metals. Ferrites are, like most other ceramics, hard and brittle. In terms of the magnetic properties, ferrites are often classified as "soft" and "hard" which refers to their low or high coercivity of their magnetism, respectively. Ferrites that are used in transformer or electromagnetic cores contain nickel, zinc, and/or manganese compounds. They have a low coercivity and are called soft ferrites. The low coercivity means the material's magnetization can easily reverse direction without dissipating much energy (hysteresis losses), while the material's high resistivity prevents eddy currents in the core, another source of energy loss. Because of their comparatively low losses at high frequencies, they are extensively used in the cores of RF transformers and inductors in applications such as switched-mode power supplies. Manganese-zinc ( $\text{Mn}_a\text{Zn}_{(1-a)}\text{Fe}_2\text{O}_4$ ) and nickel-zinc ( $\text{Ni}_a\text{Zn}_{(1-a)}\text{Fe}_2\text{O}_4$ ) ferrites are widely and commonly used.

In order to fabricate and/or synthesis aluminium based composite materials having magnetic properties, powder metallurgy is suitable and advantageous techniques due to large gaps of density and melting point between aluminium and ferrite. Mechanical alloying (MA) is one of powder

processing techniques to produce composite powders. MA is a type of high energy processing for solid-state powders, in which more than two different elemental powder particles are heavily and repeatedly deformed. Composite powder particles and/or a variety of stable and metastable nanocrystalline intermetallic compounds can be produced and/or synthesised by MA processing [1,2]. Aluminium-based composite materials fabricated from MAed powders and subsequent hot extrusions have exhibited desirable mechanical properties at room and elevated temperatures [3,4]. This is due to uniformly distributed nanosized particles together with extremely fine grained Al matrix MA and subsequent hot extrusions have exhibited desirable mechanical properties at room and elevated temperatures [3,4]. Recently, the technique of mechanochemical synthesis i.e. reaction milling (RM) [5] has attracted considerable interest. In this process, relatively thermodynamically unstable ceramics powders are decomposed by solid-state reactions within the matrix after heat treatment of mechanically activated MAed powders. Consequently, in situ formation of relatively stable dispersoids may be achieved. After producing MAed powders, a number of consolidation processes has been applied to fabricate to bulk materials, e.g. a combination of cold pressing and hot extrusion, hot pressing (HP) and hot isostatic pressing (HIP). Spark plasma sintering or synthesis (SPS) has recently attracted considerable attention [8,9]. The advantage of the SPS process is that it allows fabrication of bulk materials from MAed powders using relatively short sintering times at low temperatures.

The aim of the present work thus was to fabricate aluminium exhibiting soft magnetic properties produced by MA of pure aluminium and Ni-Zn-Cu-Zn ferrite powder followed by compaction via SPS. Mechanical and magnetic properties of the MAed powders and SPSed bulk materials were examined by hardness measurements and Vibrating Sample Magnetometer (VSM).

## 2. Experimental Procedures

### 2.1 Material preparations

Air-atomised 99.9% pure aluminium powder and ferrite (FR) powder with an average diameter of 25  $\mu\text{m}$  and 1.8  $\mu\text{m}$ , respectively was used. FR is a complex oxide, having a composition of 49.41  $\text{Fe}_2\text{O}_3$ -32.09  $\text{ZnO}$ -12.71  $\text{NiO}$ -5.79  $\text{CuO}$  (at.%). As shown in Table 1, the composition of 10 mass% FR was selected, and designation of the tested materials was included. Stainless steel balls together with 10 g of the above mixture powder and stearic acid ( $\text{C}_{17}\text{H}_{35}\text{COOH}$ ), which acted as a process control agent (PCA), were sealed in a hardened steel vial using a glove box filled with argon. The ball to powder mass ratio was approximately 7:1. MA was performed at room temperature using an SPEX8000 mixer/mill with processing time varied from 2 h to 8 h.

The MAed powders were consolidated by an SPS apparatus. Seven grams of the MAed powder was placed in a graphite die of 20 mm in diameter and 40 mm in height, and heated under vacuum with an applied pressure of 49 MPa at 873 K for 1 h.

**Table 1** Designation, starting compositions and mechanical alloying time of test materials.

Designation	Material (mass%)		MA time (h)
	matrix	compound	
12 AIFR	Pure Al	10 Ferrite	2
14 AIFR			4
18 AIFR			8

### 2.2 Material evaluations

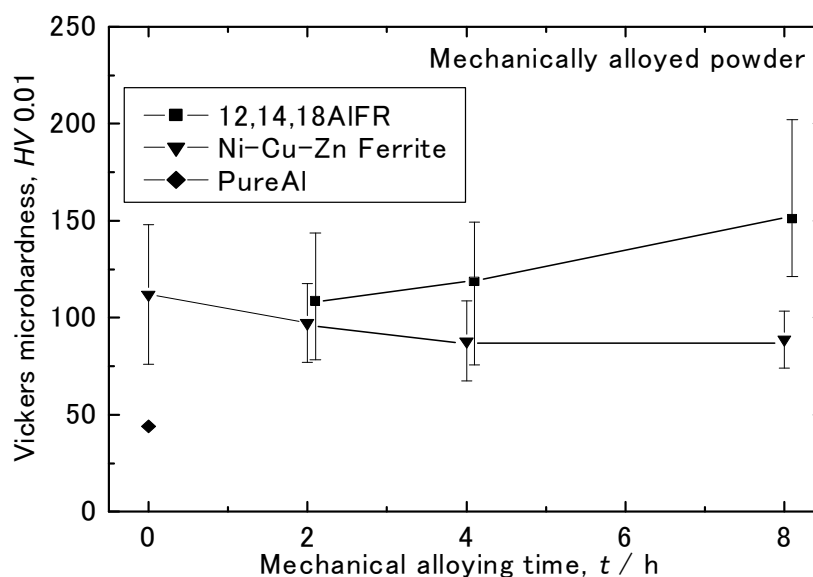
The Vickers microhardness of the MAed powders was measured with a Vickers microhardness tester using an applied load of 10 g. The average Vickers microhardness of the MAed powders was

determined from 15 particles per sample. The Vickers hardness of the SPS materials was measured with a Vickers hardness tester using an applied load of 1 kg. Density of SPS materials was measured based on the Archimedes method after covering specimens by oil wax to prevent penetration of water into pores. X-ray diffraction (XRD) analysis was performed for both the MAed powders and SPS materials using a Cu K $\alpha$  radiation source operating at 40mA and 60 kV.

Saturated magnetisation and coercive force was obtained using vibrating sample magnetometer (VSM) under magnetic field of 800 or 40 kA/m at room temperature. The size of the SPS materials for the VSM measurements was 6x4x1 mm. Saturation magnetic flux density was obtained from a multiple of saturated magnetisation and density of the SPS materials.

### 3. Results and Discussion

Figure 1 presents the change in Vickers microhardness of the Al-10 mass% FR powder together with pure FR powders as a function of different MA processing times. The hardness of the pure Al powder before MA was approximately 44 HV. The hardness of the Al-10 mass% FR powder increased gradually to over 150 HV after 4 h of MA, but a slight decrease was observed for pure FR powder down to a value of 95 HV after 8 h of MA. These results can be explained that a large amount of strain can be induced to pure Al matrix powders together with uniformly distributed FR particles. The hardness of the Al-10mass%FR powders thus increased. On the other hand, FR is ceramics that may be impossible to induce strain to FR powder during MA process, suggesting that the hardness was maintained the almost same level at the stage of 0 h of MA.

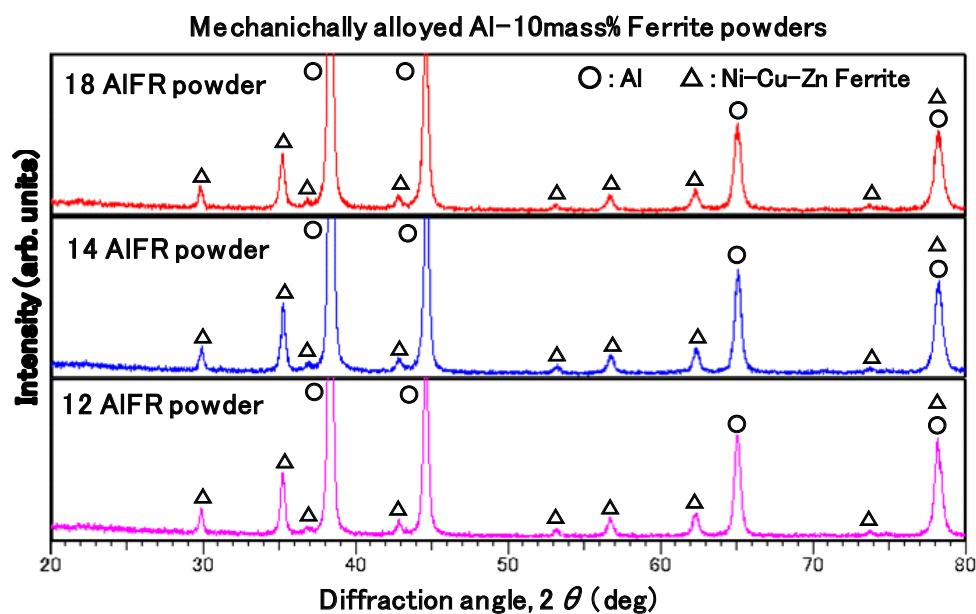


**Fig. 1** Change in the Vickers microhardness of Al-10 mass% FR powders together with FR powder as a function of mechanical alloying time.

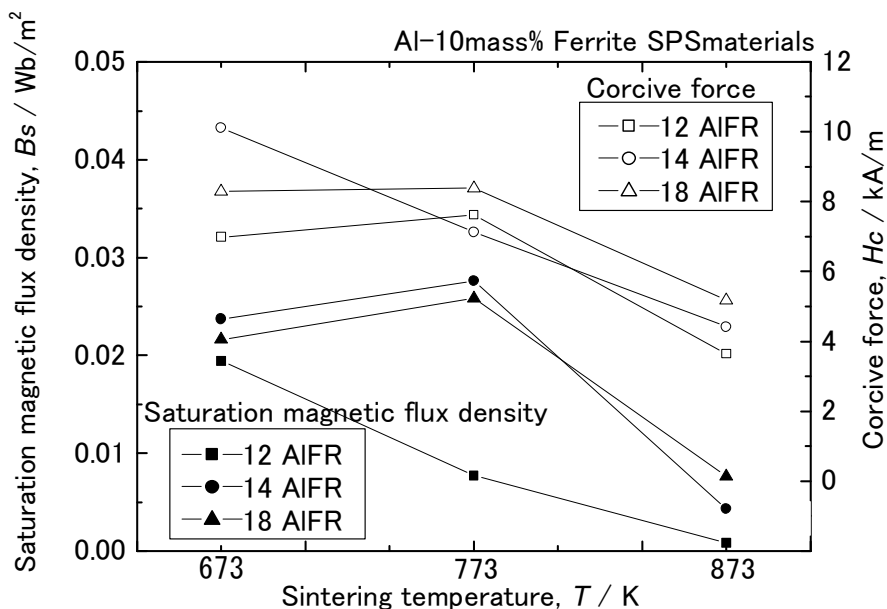
XRD patterns of Al-10mass%FR powder as a function of the MA times are shown in Fig. 2. The additive element of FR in Al matrix was detected after 2 h, 4 h and 8 h of MA, suggesting that no solid-state reaction occurred during the MA process. This clearly implies that FR particles can be dispersed uniformly into the aluminium matrix. It thus can be exhibited both reasonable magnetic properties together with relatively good mechanical properties.

Figure 3 presents changes in the saturation magnetic flux density and coercive force of for the SPS materials fabricated from 12, 14, 18 AlFR powders at different sintering temperatures. The saturation magnetic flux density of the SPS materials fabricated from 12AlFR, 14AlFR and 18AlFR powders at

both 673 K and 773 K exhibited the same value of approximately  $0.02 \text{ Wb/m}^2$ , suggesting that the MA time was not influenced on the saturation magnetic flux density. On the other hand, the saturation magnetic flux density of the SPS materials fabricated at 873 K decreased significantly due to occurring the decomposition of FR powders in the aluminium matrix during the sintering process. The coercive force for the SPS materials fabricated at 673 K exhibited a value of  $6\sim 10 \text{ kA/m}$ , and it decreased to a value of  $3\sim 5 \text{ kA/m}$  when the composite powders were SPSed at 873 K, implying that decomposition of FR phase and coarsening of grains.



**Fig. 2** X-ray diffraction patterns of Al-10 mass% FR powders with different mechanical alloying times.



**Fig. 3** Change in the saturation magnetic flux density and coercive force for the SPS materials fabricated from 12, 14, 18 AIFR powders at different sintering temperatures.

#### 4. Conclusions

- (1) The aluminium – ferrite composite powders exhibiting magnetic properties were synthesised by 2 h, 4 h and 8 h of MA process.
- (2) The bulk aluminium – ferrite composite materials were fabricated by SPS at 673 K and 773 K, minimizing solid-state reaction between aluminium and ferrite under existence of stearic acid. Magnetic properties of the bulk composite materials exhibited depending upon the addition of ferrite.

#### References

- [1] M. MIZUMOTO, A. KAGAWA and T. OHGAI: “Fabrication and Characterization of Metal Matrix Composites by Low Pressure Infiltration Method”, Reports of the Faculty of Engineering at Nagasaki University, **36**(2006)53-59.
- [2] T. MIYAJIMA, T. HONDA and Y. IWAI: Trans. Jpn. Soc. Mech. Eng, **71** (2005) 3054-3060.
- [3] T. HASEGAWA, Z. LI-PING, T. MIURA and N. NISHIWAKI: J. Jpn. Inst. Light Metals, **44** (1993) 359-364.
- [4] S. WANIKAWA, S. EMURA and M. HAGIWARA: J. Jpn. Soc. Powder Powder Metal, **46** (1998) 484-488.
- [5] K. MATSUDA, S. IKENO et al. : “Magnetic Aluminum Composites” , Japan Patent Office , Japanese Published Unexamined Patent Application No.257513 (2006).
- [6] M. Tokita : The Soc. of Powder Technology Japan, **30** (1993) 790-804.
- [7] K. MIZUUCHI, K. INOUE et al. : Proc. Jpn. Soc. Mech. Eng, **14** (2006) 87-88.
- [8] K. MIZUUCHI, K. INOUE et al. : Mater. Sci. Eng. A, **A367** (2004) 343-349.
- [9] S. YAMAMURA, H. KADOKURA et al. : J. Jpn. Soc. Powder Powder Metal, **53** (2006) 965-970.
- [10] M. KONUMA : Sanpan Jiseizairyuu, Kougakutoshokabushikigaisha (1996)53-54
- [11] M. Ma, Y. Wu, J. Zhou, Y. Sun, Y. Zhang, N. Gu : J. Magn. Magn. Mater, **268** (2004) 33-39.