Enhancing Properties of Fe-Cr-alumina Composites Prepared by Powder Metallurgy

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Abstract

Fe-based matrix composites have shown the potential for use as advanced materials for technological applications. In this study metal matrix composites (MMCs) of Fe-Cr –alumina were fabricated by powder metallurgy (PM). The effect of alumina content on the physical and mechanical properties of the composites was investigated. Scanning Electron Microscope (SEM) showed a homogeneous distribution of alumina in the matrix. Energy Dispersive X-ray (EDX) displayed the presence of Fe, Cr and alumina in the composites. Optimum condition of the composites was examined by evaluation of the parameters such as density, porosity, shrinkage, hardness, wear resistance and compressive strength. Results showed that addition of alumina greater than 5 wt. % decreased the density but increased porosity. Hardness and wear resistance of the composites increased with increasing alumina content to 20 wt. %. However, the compressive strength showed optimal value at 5 wt. % alumina.

Keywords: powder metallurgy, metal matrix composite, Fe, Cr, alumina

Introduction

To meet the requirement for devices with improved mechanical, thermal and operational properties, new materials should be developed. Composites materials that combined different materials to produce new materials with performance unattainable by the individual constituents have received attention due to technical and economical advantages worldwide.

Recently focused on Fe based matrix composite which are resistant to thermal shocks, wear and corrosion in high temperature applications has stimulated the continuous and rapid development in advanced materials. Many investigations on Fe matrix composites have been focused on Fe alloy or steel as the matrix materials (Pagounis & Lindroos, 1998, Al-Rubaie, 2000, Mukherjee, & Bandyopadhyay, 1995, Li et al, 2007). However, Fe-Cr matrix composite reinforced with alumina is still not established. When the goal is to improve wear resistance, alumina is used on account of their hardness (Vardavoulias et al, 1996). An attempt was made in this study to produce Fe-Cr matrix composites reinforced with alumina by powder metallurgy method as an alternative in choosing a wear resistance material for engineering applications.

Traditionally, wear resistance materials that was widely employed in various industries were made from solidification of castings in sand mould of Co, Ni or Fe alloy. The crystallographic structure and hardness of these materials depends on alloying and heat treatment. In the mining, cement industry and road construction, white cast iron (WCI) of hypo-to-hypereutectic composition consist of hard phases (HP) of carbide or borides embedded in a hardened metal matrix are the workhorses of wear protection. The problems with this material are solidification of castings (develop from the melt) which depends on phase equilibrium, decreases in size of HP due to sliding abrasive particles (AP) and linkage of HP which results in a brittle skeleton that promotes crack extension (Berns, 2003, Berns & Wewers, 2001).

The PM technique in the synthesis of MMCs found initial use because of the difficulty in wetting ceramic particles with molten metal. It is an important processing technique for MMCs that tend to offer homogeneity of both composition and microstructure of the matrix materials together with more control over the reinforcement distribution. This uniformity not only improves the structural properties but also the reproducibility level in the properties. The effect of alumina on the properties of the composites have been investigated and reported in this paper.

Methodology

Ferrous (Fe) powder (7.97 µm) was employed as matrix materials. Cr powder (25.60 µm) was fixed at 12 wt. % as added alloying element. Alumina powder (13.31 µm) was used as reinforcement with different loading of alumina, i.e. 0 wt. %, 5 wt. %, 10 wt. %, 15 wt. %, 20 wt. % and 25 wt. %. The composites were labeled as A0, A5, A10, A15, A20 and A25 accordingly. Powder mixtures containing 2 wt. % of stearic acid was blended for thirty minutes at 250 rpm in a drum-shaped plastic container. The mixed powder was poured into a die 10 mm in diameter and uni-axially pressed at a pressure of 750 MPa. The prepared green compacts were sintered in a vacuum furnace under 10^{-2} Torr at temperature 1373K (1100°C) for 2h with 10°C/minutes heating rate. All the parameter used in this study was determined in the preliminary study (Shamsuddin et al., 2007, Shamsuddin et al., 2008, Shamsuddin et al., 2010, Shamsuddin et al., 2011).

SEM and EDX from JEOL JSM-6460LA were used to reveal the microstructures and element compositions in The density and porosity were the composites. determined using the Archimedes principle according to ASTM B311 - 93. Prior to testing, the specimens were prepared according to standard metallographic procedure ASTM E3-95(1999). HM-114 Mitutoyo Hardness Testing Machine was used to determine the Vickers micro hardness value of the composite. The micro hardness value (HV) was determined by the size of indentation and measured at five points on the surface of the sintered composites. The average values adopted as the Vickers micro hardness of the composites. To evaluate the abrasive wear of the composites the pin on disk wear resistance test based on ASTM standard G99 was utilized. Composites obtained as pin specimens were made to slide against a stainless steel disk at a constant load of 20 N and a constant sliding velocity of 100 rpm. Wear coefficient was calculated using the Archard wear equation as shown in Eq.1.

Wearcoefficient =
$$\frac{(\text{Volumeloss, m}^3)}{(\text{Normalload, N}) \times (\text{Sliding distance,m})}$$
(1)

The compressive strength test in accordance to standard ASTM E9 was performed at room temperature on the Universal Testing Machine BISS 100kN/Hydraullic Tensile Group. A crosshead speed of 5 mm min⁻¹ was used. The deformation behavior of the composite was determined from the stress strain curve of each sample.

Results and Discussions

The effect of alumina content of the microstructure is shown in Figure 1. SEM images show uniform distribution of reinforcement at a low content of alumina. Homogeneous microstructure indicated a well mixed composite was achieved. All particles are homogeneously distributed in the mixture to obtain a good uniform microstructure (Abenojar et al, 2007). The homogeneity of a mixture has a major influence on the packing density. It was reported that the packing density increased with the homogeneity of the mixture (German, 1989).

However when the content of alumina increased it tends to form a bridge between the pairs of matrix particles and aggregates formation in the composites. A uniform distribution of reinforcement becomes impossible because of inadequate ratio of the surface areas of matrix alloy particles and reinforcement particles as shown in Figure 1(f) which presents 25 wt. % alumina amounts. Alumina particles act as a barrier against the movement of grains boundaries and hence retard grain growth. This phenomenon can be explained in Eq.2.

$$\lambda = \frac{4(1-f)r}{3f} \tag{2}$$

The volume fraction of the reinforcement (f) and its radius (r), determine the distance apart the particles (λ), and hence, prevent the movement of grain boundaries.

Figure 3 illustrates the physical properties of the composites. The average densities of 7.2542 gcm⁻³ and 4.6197 gcm⁻³ for Fe and alumina were determined by Pycnometer Density. The density of Fe is higher than alumina but decreased its bulk density when alumina was introduced in the composite. The consequent of this is leading to increased of porosity. The pores were occurred between the elements of the composites which indicated a weak interfacial bonding among the particles. When mixing was accomplished, the particles started to agglomerate which resulted in more pores and reduced the density. Agglomeration might occur during mixing stage due to cold welding at the particle contacts. As the degree of agglomeration increased the pore between agglomerates increased. The outcome is a lower packing density which

decreased in physical and mechanical properties of the composite samples (German, 1994).

Cr Fe (a) (b) (c) (d) (c) (f)

Figure. 1. SEM micrograph at varying weight percentage of alumina, (a) 0%, (b) 5%, (c) 10%, (d) 15%, (e) 20% and (f) 25%.

The presence of Fe, Cr and alumina is confirmed from the EDX spectra as shown in Figure 2.



Figure 2. EDX spectra of the composites.

During compaction process, gases are trapped by particles. Higher amount of alumina particles caused more gases to be trapped and lead to more porosities being left over in the composites. Vickers micro hardness reading demonstrated that reinforcing the composites with alumina increases the hardness value. The composites reinforced with 5 to 20 wt. % alumina particles have higher hardness value than the unreinforced samples. This can be attributed to the dispersion strengthening effect. Alumina particles with high hardness as the reinforcement phases dispersed in the matrix impeded plastic deformation. The increases in the micro hardness of the composites with increasing amount of oxides reinforcement agree well with previous findings (Rajiv et al, 1995, Mukherjee & Bandyopadhyay, 1997, Gurudu et al ,2006).

Hardness value decreased when 25 wt. % loading alumina was incorporated. This is due to reinforcement clustering that caused low wettability and more porosity that has no resistance to local plastic deformation. Higher amount of alumina particles with lower coefficient of thermal expansion (CTE) resulted in stresses due to CTE mismatch which causes more cracks and pores around the particles in the composites (Hull & Clyne, 1996).



Figure 3. The physical properties versus loading wt% of alumina.

Figure 4 shows the relationship between the wear coefficient and sliding distance of varying wt.% of loading alumina in composites. Wear coefficient increases with the increase of sliding distance. In the wear test the volume loss of the composites decreased linearly with increased sliding distance. The Fe-Cr alloy without alumina reinforcement showed highest volume loss because the metal of Fe and Cr were soft and easily wear compared to those reinforced with alumina.

For sample with 25 wt. % loading alumina showed apparent weight loss. The hardness value has

significant effect on the coefficient of wear resistance. Wear loss of the composites was decreased by increased of hardness. Higher content of alumina shows lowest wear resistance due to weak interface bonding of alumina which causes spalling of particles during wearing. This can be explained by the rule of mixture, as shown in Eq. 3.

$$\mathbf{H}_{c} = \mathbf{H}_{m}\mathbf{f}_{m} + \mathbf{H}_{r}\mathbf{f}_{r} \tag{3}$$

 H_c , H_m , and H_r are the hardness of the composite, matrix and reinforcement respectively, f_m and f_r are the volume fraction of the matrix and reinforcement respectively.

Even though the density decreased and the porosity increased with the addition of alumina particles to more than 5 wt. %, the hardness reading and the wear resistance coefficient were improved for the composites with 5 to 20 wt. % alumina particles. However, the composite's properties declined when 25 wt. % alumina particles were used. This phenomenon shows that the homogeneity of the reinforcement in the matrix plays an important part in determining the mechanical properties of this composite, apart from the porosity.



Figure 4. Wear coefficient as a function of sliding distance in loading alumina composites.

Figure 5(a) and (b) photos of composites from the side and surface views with the average dimension of 13.11 mm in length and 9.93 mm in diameter.

Figure 6 and 7 photos from side and surface views of composites with different loading of alumina after compressive strength test. All samples show a barreling effect and were crack after the test. It was observed that 25% dimension decreased experience by A25 composite sample whereas 22% for a control and other

composites about 19%. This result indicates that adding alumina as reinforcement improve the strength of the composites but the alumina amount should not exceeds 25 wt%. Sample without alumina and reinforced with 25 wt. % alumina samples were fractured due to formation of bigger cracks during loading. The causes of the cracks may arise from the clustering of alumina in the sample with 25 wt. % alumina. On the other hand the fracture in sample without alumina may due to the plastic deformation of metal without reinforcement and there were no support to prevent cracks propagation.



Figure 5. The photos of composite (a) side view and (b) surface view.



Figures 6. Photos from side view of composite samples after compressive strength test with different loading alumina, (a) 0%, (b) 5%, (c) 10%, (d) 15%, (e) 20% and (f) 25%.

Figure 8 shows compressive stress-strain curves of composites at different loading wt. % of alumina. Ultimate compressive strength was determined as the optimum values from the stress–strain curve. When stress–strain curve showed sharp-knee or a rapid drop in load, this point was determined as a fracture of the material and the test was terminated at this point. Results show that addition of 5-10 wt. % loading alumina in the composite increased the compressive

strength whereas exceeding 15 wt. % reduced to compressive strength.



Figures 7. Photos from surface view of composite samples after compressive strength test with different loading alumina, (a) 0%, (b) 5%, (c) 10%, (d) 15%, (e) 20% and (f) 25%.



Figure 8. The stress strain curve of composites loading with different wt. % alumina.

The effect of loading alumina content on compressive strength is depicted in Figure 9. The addition of alumina to 10 wt. % increases the strength. Beyond that the strength started to decline. According to Eq. 1, as the amount of alumina is increase, the distance apart the reinforcement particle decreases. Thus clustering of alumina occur which lead to lower compressive strength. The clusters activate more slip systems in the matrix to accommodate the same amount of deformation which leads to fracture of clusters at an early stage. Furthermore the presence of harder alumina particles beyond optimum amount leads to plastic incompatibility with the matrix during compressive loading leading to early crack nucleation and hence reduced ductility. Other reasons are dislocation generation due to elastic modulus mismatch and CTE mismatch between the Fe matrix and alumina reinforcement which limited the load transfer from matrix to reinforcement (Aigbodion & Hassan, 2007, Narayanasamy et al, 2008, Nguyen & Gupta, 2010, Bauri & Surappa, 2009).



Figure 9. Relationship between the compressive strength of composite as a function of alumina amount.

Conclusions

It was found that incorporating alumina in the Fe-Cr based matrix has improved the physical and mechanical properties of the composites. Composites loaded with 20wt. % leading to higher Vickers micro hardness reading and favorable wear resistance. But increasing to more than 10 wt. % alumina content decreases the compressive strength. The compressive strength of the composite achieved with the content of 5 wt. % loading alumina.

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References

Abenojar, J.F., Velasco, F and Martinez, M.A. (2007) Optimization of processing parameters for the Al + 10% B_4C system obtained by mechanical alloying. Journal of Materials Processing Technology. 184 (1-3), 441-446.

Al-Rubaie, K. S. (2000). "Equivalent hardness concept and two-body abrasion of iron-base alloys." Wear 243: 92-100.

Bauri R and M. K. Surappa. (2009). "Processing and compressive strength of Al-Li-SiCp composites fabricated by a compound billet technique," in Journal of Materials Processing Technology, 209(4), 2077-2084.

Berns, H and Wewers, B. (2001) Development of an abrasion resistant steel composite with in situ TiC particles. Wear. 251 (1-12), 179-186.

Berns, H. (2003) Comparison of wear resistant MMC and white cast iron. Wear. 24 (1-2), 47-54.

German, R.M. (1989) Particle Packing Characteristic. New Jersey: Metal Powder Industries Federation.

German, R.M. (1994) Powder Metallurgy Science. New Jersey: Metal Powder Industries Federation, 110-210.

Gurudu, R.K., Scattergood, R.O., Roch, C.C., Murty, K.L., Gurusamy, S. and McCarter, M.K. (2006) Mechanical properties of nanocrystalline Fe-Pb and Fe-Al₂O₃. Scripta Materialia. 54 (11), 1879-1883.

Hull, D. and Clyne, T. W. (1996) An introduction to composites materials. Cambridge, Cambridge University Press.

Li, J. Q., W. A. Sun, et al. (2007). "Al2O3-FeCrAl composites and functionally graded materials fabricated by reactive hot pressing." Composites Part A: Applied Science and Manufacturing 38(2): 615-620.

Mukherjee, S. K. and Bandyopadhyay, S. (1997) Mechanical and interfacial characterisation of Fe_3AI and Fe_3AI - Al_2O_3 intermetallic composite made by mechanical smearing and hot isostatic pressing." Composites Part B: Engineering. 28(1-2), 45-48.

Mukherjee, S. K. and S. Bandyopadhyay (1995). "A novel way to make Fe₃Al/Al₂O₃ composite." Materials Science and Engineering_A202: 123-127.

Pagounis, E. and Lindroos, V.K. (1998). Processing and properties of particulate reinforced steel matrix composites. Materials Science and Engineering *A*. 246 (1-2), 221-234.

Q. B. Nguyen and M. Gupta. (2010). "Enhancing compressive response of AZ31B using nano-Al₂O₃ and copper additions," in Journal of Alloys and Compounds, 490(1-2), 382-387.

R. Narayanasamy, V. Anandakrishnan and K.S. Pandey. (2008). "Effect of carbon content on instantaneous strain-hardening behaviour of powder metallurgy steels," in Materials Science and Engineering: A, 497(1-2), 505-511.

Rajiv, E. P., Iyer, A. and Seshadri, S.K. (1995) Tribological properties of cobalt-partially stabilized zirconia (PSZ) composites in dry sliding conditions. Wear 189(1-2), 100-106.

Saidatulakmar Shamsuddin, Shamsul Baharin Jamaluddin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2007). "Compaction study of particulate iron-chromium matrix composite reinforced with alumina." Proceeding of the National Metallurgical Conference, Malaysia.

Saidatulakmar Shamsuddin, Shamsul Baharin Jamaluddin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2007). "Microstructure and Hardness Properties of Fe-Cr-Al₂O₃ Composites." Malaysian Journal of Microscopy, 4, 118-123.

Saidatulakmar Shamsuddin, Shamsul Baharin Jamaluddin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2008) "Characterization of Fe-Cr-Al₂O₃ Composites Fabricated by Powder Metallurgy Method with Varying Weight Percentage of Alumina." Journal of Physical Science, 19(1), 89-95.

Saidatulakmar Shamsuddin, Shamsul Baharin Jamaluddin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2007) "Preliminary study of iron-chromium matrix composite reinforced with alumina," in Solid State Science and Technology Letters, 16 (1 &2), 38-43.

Saidatulakmar Shamsuddin, Shamsul Baharin Jamaluddin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2010). "Influence of Binder in Iron Matrix Composites." American Institute of Physics Conference Proceedings, 1217, 419-423.

Saidatulakmar Shamsuddin, Shamsul Baharin Jamaluddin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2011). "Heating Rate Effects on Properties of Powder Metallurgy Fe-Cr-Al₂O₃ Composites." Advanced Materials Research Journal, 173, 84-89.

Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2008). "The Characteristics of vacuum sintered Fe-Cr-Al₂O₃ Composites." Proceeding of the 24th Regional Conference on Solid State Science and Technology, Malaysia.

Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2008). "Effect of Particle Size on Fe-Cr-Al₂O₃ Metal Matrix Composite," in Proceeding of Malaysian Metallurgical Conference, Malaysia. Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2010). "The effects of Al_2O_3 amount on the microstructure and properties of Fe-Cr matrix composites." Metallurgical and Materials Transactions A, 41A, 3452-3457.

V. S. Aigbodion and S. B. Hassan. (2007). "Effects of silicon carbide reinforcement on microstructure and properties of cast Al-Si-Fe/SiC particulate composites," in Materials Science and Engineering: A, 447(1-2), 355-360.

Vardavoulias, M., M. Jeandin, et al. (1996). "Dry sliding wear mechanism for P/M austenitic stainless steels and their composites containing Al2O3 and Y2O3 particles." Tribology International 29(6): 499-506.