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Microstructure and Hardness of TiC Particle-reinforced Fe Self-fluxing Alloy Powders Based Hybrid Composite Prepared by High Energy Ball Milling

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Abstract The Fe-based self-fluxing alloy powders and TiC particles were ball-milled and subsequently compacted and sintered at various temperatures, resulting in the TiC particle-reinforced Fe self-fluxing alloy hybrid composite, and the microstructure and micro-hardness were investigated. The initial Fe-based self-fluxing alloy powders and TiC particles showed the spherical shape with a mean size of approximately 80 μm and the irregular shape of less than 5 μm, respectively. After ball-milling at 800 rpm for 5 h, the powder mixture of Fe-based self-fluxing alloy powders and TiC particles formed into the agglomerated powders with the size of approximately 10 μm that was composed of the nano-sized TiC particles and nano-sized alloy particles. The TiC particle-reinforced Fe-based self-fluxing alloy hybrid composite sintered at 1173 K revealed a much denser microstructure and higher micro-hardness than that sintered at 1073 K and 1273 K.

Keywords: Fe-based self-fluxing alloy powder, TiC particles, Hybrid composite, High energy ball milling, Microstructure, Micro-hardness

1. Introduction

Fe-based self-fluxing alloy containing a large amount of Cr, significant amounts of Si, B and some amount of C to lower the melting point of the alloys down to 1000°C has been used for the post-spray fusing treatment. During the fusing treatment, the self-fluxing alloy coating becomes partially molten so that the oxides formed during spraying can be removed by the fluxing action of Si and B, the internal porosities are omitted, and the adhesion to the substrate is improved [1]. However, much higher mechanical properties such as wear resistance depending on the applications are often demanded in the self-fluxing alloys. In order to improve such properties, therefore, it is very useful to fabricate the metal matrix composites (MMC) reinforced with a second phase, such as ceramic oxides, nitrides and carbides. In general, there are several routes processing including infiltration methods, squeeze casting and powder metallurgy to fabricate the MMC [2]. Powder metallurgy is particularly the most preferred one because of the ability producing the near net-shaped components, easy control of the reinforcement volume fraction and suppression of chemical reaction at the interface between matrix and reinforcement. Among the powder metallurgy methods, the high energy ball milling is an effective unconventional technique currently used in inorganic materials synthesis and processing to obtain the nano-sized materials [3]. It consists of repeated events of energy transfer, promoted by the milling device, from the milling tools (generally balls) to the milled powder. On the other hand, the kind and shape of the reinforcement phase play a very important role in the final properties of the composite [4]. In particular, SiC and TiC particles have been used as reinforcement because of relatively high physical and mechanical properties [5, 6]. Under this back-

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ground to improve the mechanical property of the self-fluxing alloy coating formed during spraying, we recently applied the high energy ball milling for fabrication of Fe-based self-fluxing alloy powders based hybrid composites reinforced with SiC particles, and reported their microstructural characteristics and micro-hardness [7].

The target in this study is to fabricate the Fe-based self-fluxing alloy powders based hybrid composite reinforced with TiC particles by the high energy ball milling and subsequent sintering, and to investigate its microstructure and micro-hardness.

2. Experimental Procedure

The initial Fe-based self-fluxing alloy powders contain 3.89 wt% B, 3.51 wt% C, 3.84 wt% Cr, 2.12 wt% Si, 0.72 wt% V elements as previously analyzed [7]. In general, the B and Cr elements are added to improve the hardenability of iron and to enhance the oxidation resistance, respectively. The Fe-based self-fluxing alloy powders and TiC particles of 25% in volume fraction were ball-milled under a high purity argon atmosphere using 5 mm stainless steel balls. The ball to powder weight ratio was 20:1 and the rotation rate was 800 rpm for 5 h. The powder mixtures were collected in the glove box filled with argon gas, and compacted under 800 MPa for 10 min and sintered at relatively high temperature at 1073 K, 1173 K and 1273 K for 3 h. The microstructure of powders and hybrid composites obtained was characterized by a scanning electron microscope (SEM) and a transmission electron microscope (TEM). The powder size and shape were investigated by an image analysis. Micro-hardness was measured by a Vickers micro-hardness tester under a load of 500 g for 15 sec.

3. Results and Discussion

The typical SEM photographs of initial Fe-based self-fluxing alloy powders and TiC particles are shown in Fig. 1a) and b), respectively. The initial Fe-based self-fluxing alloy powders were spherical in shape, whereas the TiC particles showed irregular shape. After milling, the powder mixture of Fe-based self-fluxing alloy powders and TiC particles showed again the spherical shape as shown

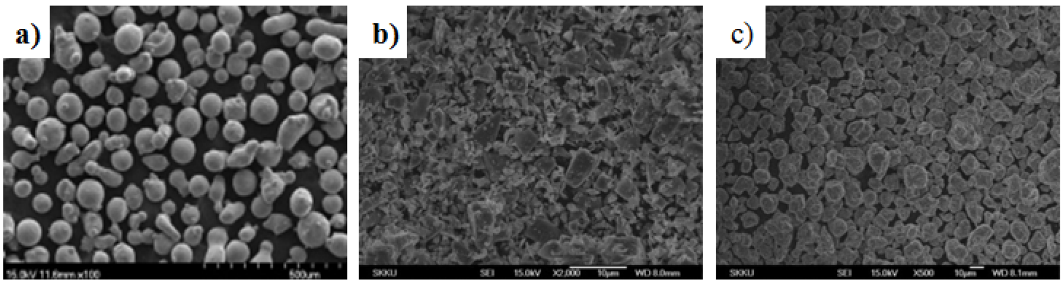

Fig. 1. SEM images showing initial Fe-based self-fluxing alloy powders (a), TiC particles (b) and as-milled Fe-based self-fluxing alloy powders with TiC particles (c).

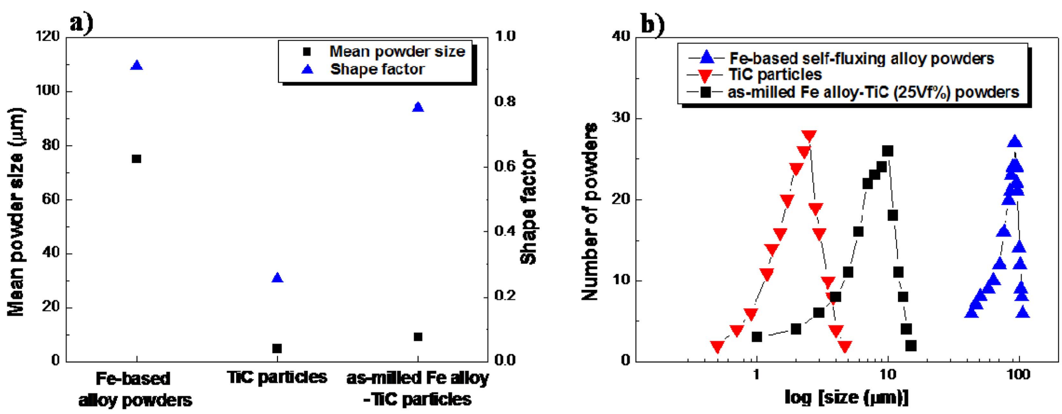

Fig. 2. (a) Mean powder size and shape factor and (b) size distribution curves of initial Fe-based self-fluxing alloy powders, TiC particles and as-milled Fe-based self-fluxing alloy powders with TiC particles.

in Fig. 1c). The size, shape factor and size distribution obtained from Fig. 1 are quantitatively shown in Fig. 2. The initial Fe-based self-fluxing alloy powders and TiC particles had the average size of approximately 80 μm and less than 5 μm, respectively. The size of powders in the powder mixture with TiC particles was approximately 10 μm after milling, which is much larger than that of the as-milled Fe-based self-fluxing alloy powders showing approximately 2 μm [7]. The shape factors of initial Fe-based self-fluxing alloy powders and powders in the powder mixture were more than 0.8, indicating that the powders were almost sphere. However, the TiC particles had the shape factor of less than 0.3. The size distribution of Fe-based self-fluxing alloy powders was inbetween 43 and 107 μm, and that of TiC particles was inbetween 8 and 61 μm. The size distribution of powders in the powder mixture showed 1~15 μm. The highest number peak of powders corresponded to the average size of powders.

Fig. 3 shows TEM photographs of high energy ball-milled powder mixture of Fe-based self-fluxing alloy powders and TiC particles. For TEM observation, the ultrasonic method was performed for the dispersion of the agglomerated powders in the powder mixture. The agglomerated powders with a mean size of approximately 10 μm as shown in Fig. 1 and 2 were composed of nano-sized alloy particles and TiC particles as shown in Fig. 3a) and b), respectively. The particle size in alloy powder mixture was approximately 60 nm, which is almost same with that in the powder mixture of Fe-based self-fluxing alloy powders and SiC particles [7]. However, the particle size of TiC was approximately 80 nm. From the results of Fig. 1c) and Fig. 3, it could be understood that the nano-sized TiC particles were embedded into the agglomerated powders consisting of the nano-sized alloy particles during milling.

Fig. 4 shows SEM photographs of the TiC particle-reinforced Fe-based self-fluxing alloy hybrid composites sintered at 1073 K, 1173 K and 1273 K. After sintering at lower temperature, 1073 K, there existed still the agglomerated powders and many voids, indicating that sufficient substance migration including diffusion had not occurred during sintering. Additionally, at the highest sintering temperature, 1273 K, in this study, the TiC particle-

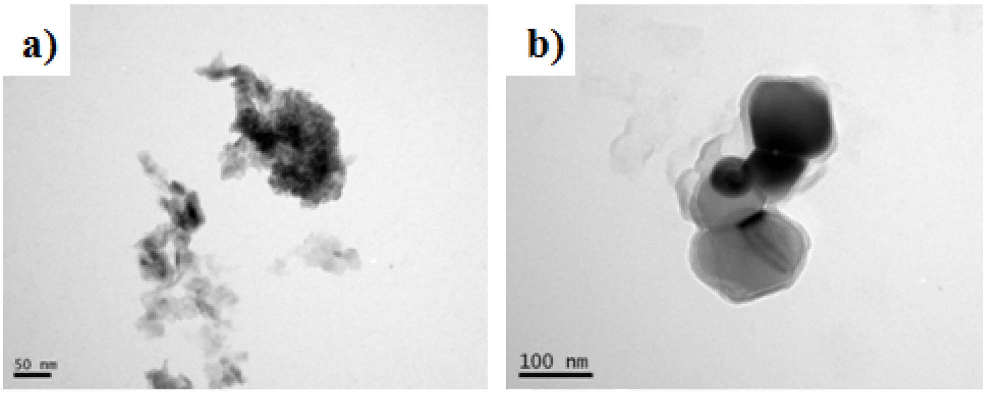

Fig. 3. TEM images of as-milled Fe-based self-fluxing alloy powders (a) and TiC particles (b).

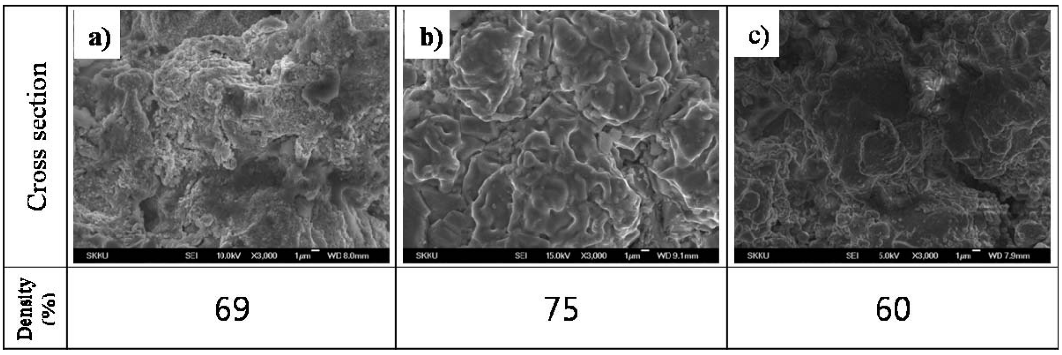

Fig. 4. SEM photographs and relative density of the TiC particle-reinforced Fe-based self-fluxing alloy hybrid composite sintered at 1073 K (a), 1173 K (b) and 1273 K (c).

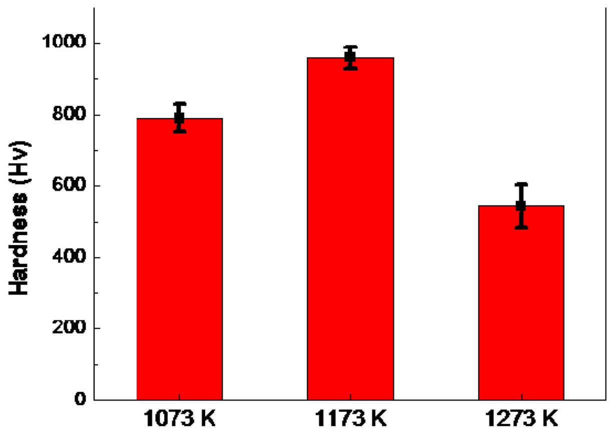

Fig. 5. Micro-hardness of the TiC particle-reinforced Fe-based self-fluxing alloy hybrid composite sintered at 1073 K, 1173 K and 1273 K.

reinforced Fe-based self-fluxing alloy hybrid composite showed sufficiently inhomogenized surface with many defaults. However, the TiC particle-reinforced Fe-based self-fluxing alloy hybrid composite sintered at 1173 K revealed relatively well-homogenized and bonded microstructure between the agglomerated powders. This microstructure characteristics well correspond to the measured density. The TiC particle-reinforced Fe-based self-fluxing alloy sintered at 1173 K showed the highest relative density of 75%, whereas the samples sintered at 1073 K and 1273 K had approximately 69% and 60%, respectively. However, there was no change in phases during sintering at different temperatures even though the result of XRD analysis was not shown.

The micro-hardness of the TiC particle-reinforced Fe-based self-fluxing alloy hybrid composites sintered at 1073 K, 1173 K and 1273 K is shown in Fig. 5. It has been previously reported that, after sintering at 1073 K, the micro-hardness of Fe-based self fluxing alloy was approximately 600 H_v and the Fe-based self-fluxing alloy hybrid composite reinforced with SiC particles of 25 wt% had the micro-hardness of approximately 700 H_v [7]. However, as shown in Fig. 5, the micro-hardness of the TiC particle-reinforced Fe-based self-fluxing alloy hybrid composites sintered at 1073 K was approximately 800 H_v. In general, the TiC particles have the micro-hardness of approximately 2900 H_v [6], which is higher than the SiC particles with the micro-hardness of approximately 2000 H_v [5]. It is therefore considered that the TiC particle-reinforced Fe-based self-fluxing alloy hybrid composites sintered at 1073K showed higher micro-hardness than the Fe-based self-fluxing alloy matrix and the SiC particle-reinforced Fe-based self-fluxing alloy hybrid composites, despite that the matrix and hybrid composites reinforced with SiC particles had much more homogeneous sintering microstructure and higher relative densities of 87% and 83%, respectively [7], compared to the TiC particle-reinforced Fe-based self-fluxing alloy hybrid composite showing the relative density of 69%. On the other hand, the TiC particle-reinforced Fe-based self-fluxing alloy hybrid composite sintered at 1173K showed the highest micro-hardness, whereas, at highest sintering temperature, 1273 K, its micro-hardness was very low. This results well correspond to the microstructural features and relative density shown in Fig. 4.

4. Conclusions

The Fe-based self-fluxing alloy powders and TiC particles were successfully mixed by the high energy ball milling at 800 rpm for 5 h, subsequently compacted under a pressure of 800 MPa for 10 min and sintered at 1073 K, 1173 K and 1273 K for 3 h to obtain the TiC particle-reinforced Fe-based self-fluxing alloy hybrid composite. After ball-milling, the agglomerated powders with the size of approximately 10 μm consisting of the nano-sized TiC particles and the nano-sized alloy particles were obtained from the initial alloy powders showing the spherical shape with a mean size of approximately 80 μm and the irregular shaped TiC particles of less than 5 μm. The micro-hardness of TiC particle-reinforced Fe-based self-fluxing alloy hybrid composite was much higher than that of both Fe-based self-fluxing alloy matrix and Fe-based self-fluxing alloy hybrid composite with SiC particles. In particular, the TiC particle-reinforced Fe-based self-fluxing alloy hybrid composite sintered at 1173 K showed the densest microstructure and the highest micro-hardness.

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