DENSIFICATION AND MECHANICAL PROPERTIES OF FEMN13-TiC COMPOSITES FABRICATED BY PULSED ELECTRIC CURRENT SINTERING PROCESS

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Received: 01.11.2018
Accepted: 26.11.2018

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Abstract

In the present work, FeMn13-40 wt.% TiC composites was fabricated by pulsed electric current sintering (PECS) process at different temperatures between 990 and 1020°C under a pressure of 60 MPa with a holding time of 5 min in the vacuum. Phase identification was done using the X-ray diffraction. The relative density, the microstructure and the hardness of the samples were characterized. The results showed that the relative density of FeMn13-TiC composites increased with the increase of sintering temperature. The lowest porosity (3.84%) and the highest hardness (70.54 HRC) of the sample were achieved by PECS process, namely sintering at the temperature of 1020°C under the applied pressure of 60 MPa for 5 min.

Keywords: FeMn13-TiC composites, PECS, relative density, hardness, sintering

1 Introduction

Nowadays, the demand for metal matrix composites has become a necessity in the structural engineering field, due to their attractive physical and mechanical properties. High manganese steel (HMS) exhibits both excellent toughness and hardenability under high impact conditions [1, 2]. Hence, it has been widely used in specific applications such as rail tracks, dredge buckets, hammer crushers and a variety of high impact and wear-resistance operations [3-5]. Among various ceramic particles, titanium carbide-TiC is expected to combine with HMS to enhance the hardness and the wear-resistance of HMS. Owing to its high hardness and high wear-resistance, TiC was possible to become reinforcement with high wettability and thermodynamic stability in molten Fe [7, 8]. Recently, many researchers have studied the materials which are relevant to the FeMn13-TiC composite. This composite was fabricated by several routes which were classified into liquid state process (molten/casting method) and solid-state process such as powder metallurgy, mechanical alloying and carbon-thermal reduction method [9-15, 19, 20, 24]. Sanjabi et al. fabricated TiC-316 stainless steel nanocomposites at 1400oC with the highest

DOI 10.12776/ams.v24i4.1195 p-ISSN 1335-1532
e-ISSN 1338-1156

hardness of 67 HRC by vacuum sintering method [9]. Besides, Li et al. produced in-situ TiC-iron matrix composites utilizing ferrotitanium and black carbon powders with a combination of in-situ and spark plasma sintering (SPS) process. The hardness of the sintered sample reached 64 HRC [11]. Particularly, Wang et al. gave a good inclusive review of different sintering processes of HMS-TiC composites. The hardness of the sintered samples was about 69 HRC even in different processes such as vacuum sintering, hot pressing, microwave sintering and spark plasma sintering [12]. The results pointed out the pulsed electric current sintering (PECS, also called as spark plasma sintering) process gives a comprehensive mechanical property within a shorter time and lower sintering temperature. As far as concerned, PECS has been considered to be one of the latest promising processes to consolidate advanced materials with a high density and minimal grain growth [6, 12-17, 19]. However, the effect of the sintering parameters, especially, the sintering temperature on the mechanical properties of FeMn13-TiC composites produced by PECS process has not been reported yet. In order to discuss the fabrication of FeMn13-TiC composites by PECS process, the sintering temperature is one of the most important factors. In this study, the densification and the mechanical properties of FeMn13-TiC composites produced by PECS process with different sintering temperatures are investigated.

2 Experimental procedures

The starting powder of FeMn13 with the average particle size of 20 μm and its chemical composition shown in **Table 1** was prepared by high energy ball-milling in previous work [18]. Commercial TiC powder (Changsha Langfeng metallic material Co. Ltd, China) with the average particle size of 4 μm and the purity of 99.6 % was used as reinforcement material.

Table 1 The chemical composition of the FeMn13 powder

Element	Mn	C	Fe	Cr	Ni	Others
wt.%	13.42	0.96	82.26	2.20	0.39	Bal.

The FeMn13 powder was mechanically mixed with 40 wt.% of TiC powder by high energy ball milling (Planetary Mill Pulverisette 6, Germany) for 5 h with the ball-to-powder ratio of 10:1. Subsequently, the mixed powders were put into a cylindrical graphite die (ϕ30x15.4x30mm) with a ϕ1.8 x 3 mm hole for temperature measurement by a thermocouple. The sintering process of compacted powder was conducted by PECS apparatus (LABOX 210, Sinterland Co. Ltd, Japan) at sintering temperature ranging from 990 to 1020°C for 5 min with heating rate of 50°C /min under an applied pressure of 60 MPa in the vacuum.

The porosity was calculated using the liquid replacement method in distilled water. The phases were identified by an X-ray diffractometer (XRD, D8 Advance, Germany). A scanning electron microscope (SEM, JSM-6510LV, Japan) was used to observe the surface morphology of the samples. The hardness and deformation test were performed by a Rockwell hardness tester (ATK 600, Mitutoyo, Japan) and a universal testing machine at room temperature (UTM, DZ-WAW600, China). The relative deformation of sintered samples was determined through the compressive strength test under a load of 900 kN.

3 Results and discussion

Fig. 1 presented the XRD patterns of the as-mixed powder and sintered samples by PECS process at different temperatures ranging from 990 to 1020°C. These patterns showed two main

DOI 10.12776/ams.v24i4.1195 p-ISSN 1335-1532
e-ISSN 1338-1156

peaks of the α-Fe solution, which correspond to (110) and (200) diffraction planes. And the TiC phase was well defined with five main peaks, which correspond to (111), (200), (220), (311) and (222) diffraction planes. The reports of Wang and Rong also obtained the same results of TiC and α-Fe solution phases existed in the composite [12, 20]. In addition, the patterns indicated that after temperature reaching 1020°C, the phase composition was not changed, and no impurities was found. It means that there was no reaction or phase transformation during the sintering process. However, there was no phase related to the Mn presented in the XRD patterns, hence the Mn atoms were completely dissolved into steel lattice as the substitution atoms after sintering process. It was caused by the strain in the α-Fe lattice and slight dislocation of the peaks of α-Fe phase to the left compared to the α-Fe standard and the starting powder pattern. This could be demonstrated that the temperature smaller than 1020°C did not affect the phase transformation of FeMn13-TiC composite.

Fig. 2 illustrated the porosity of the sintered samples as a function of the sintering temperature. The porosity decreased considerably with an increasing of temperature. The sample sintered at 1020°C had the lowest porosity of 3.84 %. It could be explained that the diffusivity of atom became stronger when the temperature increased.

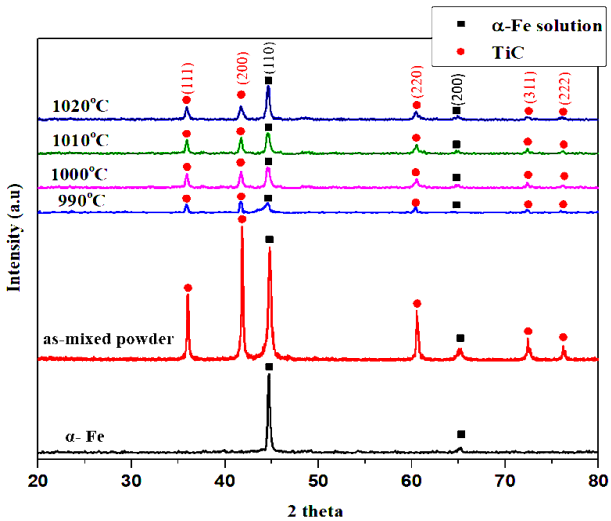

Fig. 1 XRD patterns of the as-mixed powder and sintered samples

Furthermore, the more amount of contact points between the particles formed at a high temperature activated the transport of matter leading to a high number of necks between particles. The reason might come from motivation the diffusion and evaporation-condensation of the matter on the surfaces with consequent bulk densification. Meanwhile, if the samples were sintered at the sintering temperature higher than 1020°C which caused severe volatilization of metal due to the vacuum sintering, the steel matrix would be melted and squeezed out of the mould, resulting in the continuous increase of the displacement and the formation of voids in the sample [21]. The temperature played an important role in forcing the sintered sample to become a dense bulk material.

DOI 10.12776/ams.v24i4.1195 p-ISSN 1335-1532
e-ISSN 1338-1156

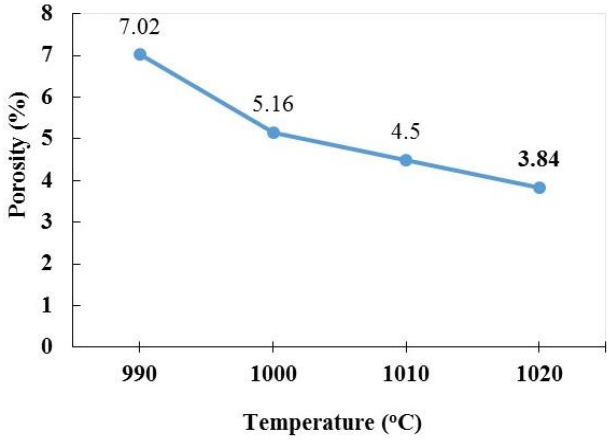

Fig. 2 The effect of the sintering temperature on the densification of FeMn13 - TiC composites

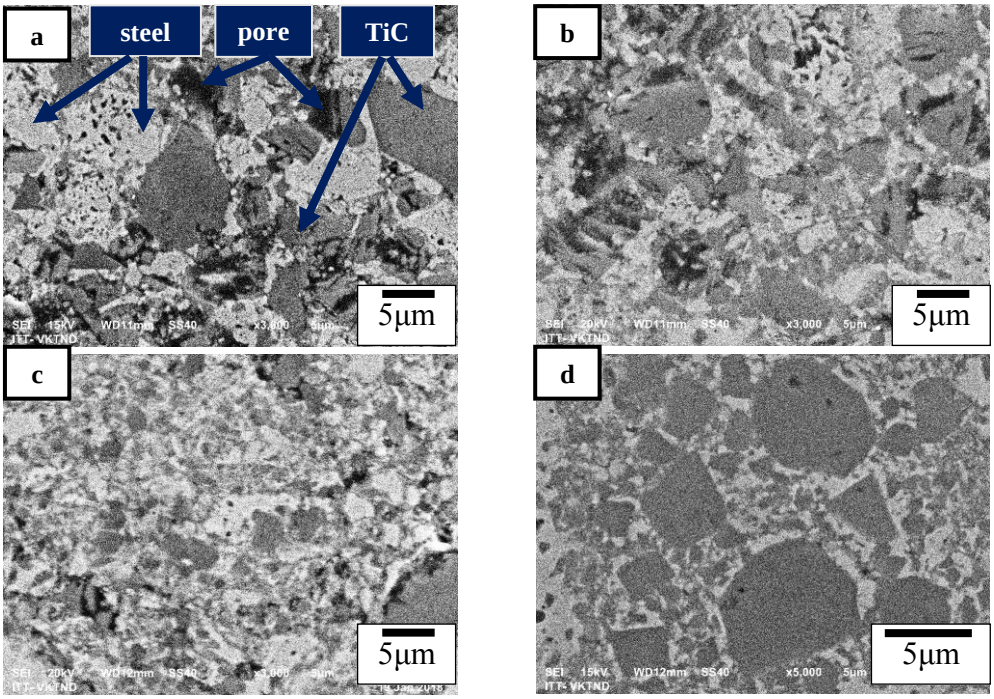

Fig. 3 Microstructure images of polished samples sintering at different sintering temperatures a. 990°C, b. 1000°C, c. 1010°C, d. 1020°C

Fig. 3 depicted the SEM images of the polished surface of the sample sintered at different temperatures ranging from 990 to 1020°C. There were three phases existed in the samples that included the grey, bright, dark regions corresponding to the TiC, steel phases, and pores, respectively. In **Fig. 3a** (at 990°C), the sintered sample had high porosity, the pores

DOI 10.12776/ams.v24i4.1195 p-ISSN 1335-1532
e-ISSN 1338-1156

agglomerated in bulk and existed inside TiC particles. It could be started as an initial crack from inside the sample. It was revealed that the diffusion process was not completed with sample sintering at 990°C. Moreover, it caused the discontinuity of the steel matrix and the decrease in the bonding between particles. Meanwhile, at the higher temperature (**Fig. 3b**, **c**, and **d**), the pores reduced considerably both their quantity and size, indicating the high densification. Especially, **Fig. 3d** (at 1020°C) showed the well-defined grain boundaries between TiC particles and steel matrix. There was no gap between reinforcement and steel matrix, which led to a better performance and minimized the failure appearance during the deformation. The number of pores was reduced because the individual grains came closer to each other due to the higher driving force during sintering at higher temperature. The results also showed the grain coarsening of TiC with the raising of temperature. It could be said that the pores reduced and the bonding between the reinforcement and matrix of the composite enhanced with the increasing of the temperature.

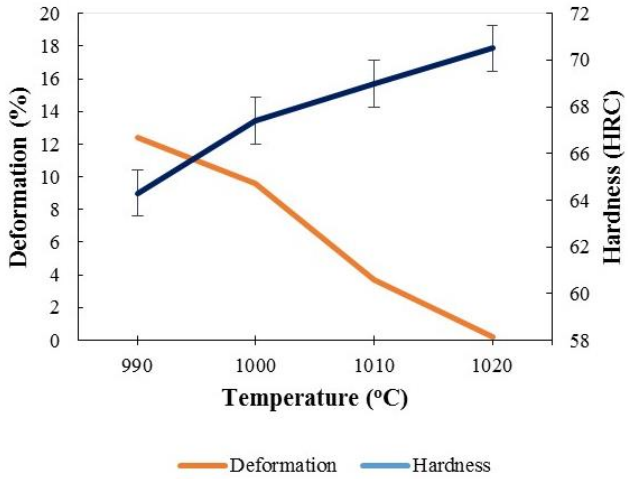

Fig. 4 The effect of sintering temperature on the mechanical properties of FeMn13-TiC composites with different sintering temperatures

Fig. 4 displayed the effect of the sintering temperature on the mechanical properties of FeMn13-TiC composites sintered at different sintering temperatures ranging from 990 to 1020°C. The hardness of the sintered samples was inversely proportional to their deformation property and linearly proportional to the increase of the sintering temperature. The reason might be that the hardness is a property of the resistance to localize plastic deformation, and the higher hardness is, the less deformation is. These results corresponded to the densification results as shown in **Fig. 2**. The sample sintered at 1020°C had the highest hardness of 70.54 HRC and the lowest deformation of 0.21 %. The enhancement of the sample hardness consolidated at 1020°C caused by the high densification process and the elimination of pores in the microstructure of composites. Moreover, the grain coarsening occurred by the higher temperature led to the hardness reduction following the Hall-Petch relationship [22, 23]. In comparison, the hardness of 50 wt.% TiC-465 stainless steel composites sintered at 1400°C [24] was lower than that of obtained FeMn13-TiC composites in the present study. Meanwhile, Wang et al. have successfully fabricated HMS-50 wt.% TiC composites using various processes that includes

DOI 10.12776/ams.v24i4.1195 p-ISSN 1335-1532
e-ISSN 1338-1156

PECS process at the higher temperature (over 1300°C) achieved the highest hardness of about 70 HRC [12]. Therefore, the temperature has significantly affected the hardness and the deformation properties of FeMn13-TiC composites produced by PECS process. It was also suggested that the PECS process can be used to consolidate these FeMn13-TiC composites to the high density with high mechanical properties.

4 Conclusions

FeMn13-TiC (40 wt.%) composites has been successfully fabricated by PECS process. The sintering temperature had a significant effect on the porosity and the mechanical properties of samples. The densification, the hardness, and the strength were enhanced with an increasing of the sintering temperature. When the sample was sintered at 1020°C for 5 min, the lowest porosity, the lowest deformation and the highest hardness of 3.84 %, 0.21 %, and 70.54 HRC, respectively, were achieved.

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Acknowledgments

This research was funded by the Ministry of Education and Training of Vietnam through Project number B2016-BKA-23. The authors would also like to thank Hanoi Pedagogical University 2 for financial support with grant number C.2018-18-06.