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Effect of warm compaction and microwave sintering on the densification, tribological and electrochemical properties of TiC/316L composites

Abstract: In this study, TiC/316L stainless steel composites were fabricated by combining warm compaction and microwave sintering (WMS). X-ray diffraction analysis was used to identify the phases in the material. Microstructure characteristics of composites were performed by means of scanning electron microscopy (SEM). Density, microhardness, wear resistance, and corrosion resistance were evaluated at room temperature. Results revealed that TiC/316L composites with higher relative density and finer properties can be obtained by WMS, compared with conventional powder metallurgy process. For all the tested species, the hardness of the composites prepared by WMS is higher than that of the composites prepared by conventional powder metallurgy (P/M). Wear tests showed that the WMS species exhibited significantly good wear resistance. Furthermore, it is found that the composites prepared by WMS were not only has high mechanical properties but also better corrosion resistance compared with the conventional P/M species.

Keywords: powder metallurgy; properties; stainless steel; titanium carbide.

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1 Introduction

Powder metallurgy (P/M) is thought to be the most common preparation technique for ceramic particles reinforced metal matrix composites (MMCs) because it provides a uniform distribution of ceramic particles in the composites [1–3]. However, P/M composites have relatively poor mechanical properties owing to their low relative density when they are obtained by conventional methods [4–6]. Thus, it is important for any P/M composites to have a high densification so that mechanical properties can be increased [7]. Previous literatures have reported that the higher densification of P/M composites can be achieved by modifying the compression methods and/or sintering methods of P/M process [8–10].

Generally, the process of compression is carried out by a conventionally cold compaction method. Compared with cold compaction, warm compaction can obtain green compacts with higher relative density under a lower pressure [11]. On the other hand, as conventionally sintered P/M materials exhibit numerous internal porosities [12], microwave sintering offers unique advantages, which include saved energy, enhanced densification, and suppressed grain growth owing to very rapid heating rates and cycles [9, 10]. Therefore, the combination of warm compaction and microwave sintering (WMS) as a preparation process of P/M composites has a good potential for enhanced densification and mechanical properties.

This work presents the effects of WMS on the properties of TiC/316L stainless steel composites. For purpose of comparison, conventional P/M TiC/316L composites were also prepared, and the microstructure and properties of the different species were comparatively investigated.

2 Experimental

2.1 Preparation of materials

Commercial 316L stainless steel powders (from CISRI, Beijing, China) with particle size ranging 25–30 μm and TiC powders (from Zhuzhou Cemented Carbide Group Co. Ltd., Zhuzhou, China) with particle size ranging 3–5 μm were used as starting materials for preparing TiC/316L composites. Mixing of the 316L powders with 5 wt.% TiC powders, was carried out by dry-mixed process in stainless steel containers for 120 min. The preparation process of WMS can be described as follows: at the first step, mixed powders are preheated in a heated die at 130°C and compressed at 500 MPa to obtain the green compact. Lubricant, 1 wt.% was used to minimize friction during compacting operations. The green compacts were sintered in a microwave furnace (HAMilabV3, Changsha, China) with a heating rate of 45°C/min and a holding time of 15 min at 1250°C. In order to have an integrated comparison, conventional P/M species were also prepared by cold compaction at 500 MPa and vacuum sintering with a heating rate of 5°C/min and a holding time of 60 min at 1250°C.

2.2 Characterization

Density was measured by the Archimedes' method with an immersion medium of water. On an average, five species were taken for density measurement. Hardness was measured on polished surfaces using a Vickers microhardness tester (HXS-1000AK, China) with an applied load of 100 gf and a dwell time of 20 s.

Wear behaviors were measured by a block on ring tribotester (MM200, China) using a rotating speed of 240 r/min and a load of 100 N. The sliding velocity was 0.5 m/s. The distance of the test was 1000 m. The counterpart was a GCrl5 steel ring with hardness of HRC 60. All of the wear tests were conducted at room temperature (25°C) in air. The weight loses were obtained from the differences in weight of the block species measured before and after the tests. The weight loss was converted into volume loss values used of sintered density of sample. For each test condition, at least three tests were performed, and the average was used.

Electrochemical measurements were performed using an AUTOLAB potentiostat (PCI4/750, China) with the general purpose electrochemical software (GPES). The polarization curves were measured at a scan rate of 0.5 mV/s from the initial potential of -200 mV vs. open-circuit potential, which were measured after 10 min immersion before tests, to the final current density of 1 mA/cm^2. Species areas of 2.0–2.5 cm^2 were immersed in the solution. The saturated calomel electrode (SCE) and Pt were the reference and counting electrodes, respectively. The experiments were conducted in a 3.5 wt.% NaCl aqueous solution at room temperature (25°C).

The immersion tests were carried out by suspending the rectangular species (15×15×3 mm) in a still solution of 15 wt.% H_2SO_4 exposed to atmospheric air for 8 days. Acid solutions were renewed every 2 days. The results of the corrosion tests were evaluated by mass loss measurements.

After the tests, species surfaces were investigated by scanning electron microscope (FEI Quanta 200, Holland).

3 Results and discussion

3.1 Microstructure and X-ray diffraction analysis

Figure 1 shows the optical micrograph of composites containing 5 wt.% TiC. In the conventional P/M species (Figure 1A), a considerable amount of spherical grain existed, and the pore structure was irregular, indicating that the species was not appropriately sintered. The species prepared by WMS showed a homogeneous structure, which was beneficial to the improvement of mechanical properties due to uniform and volumetric heating in the microwave field [13]. Moreover, the microstructure of the species

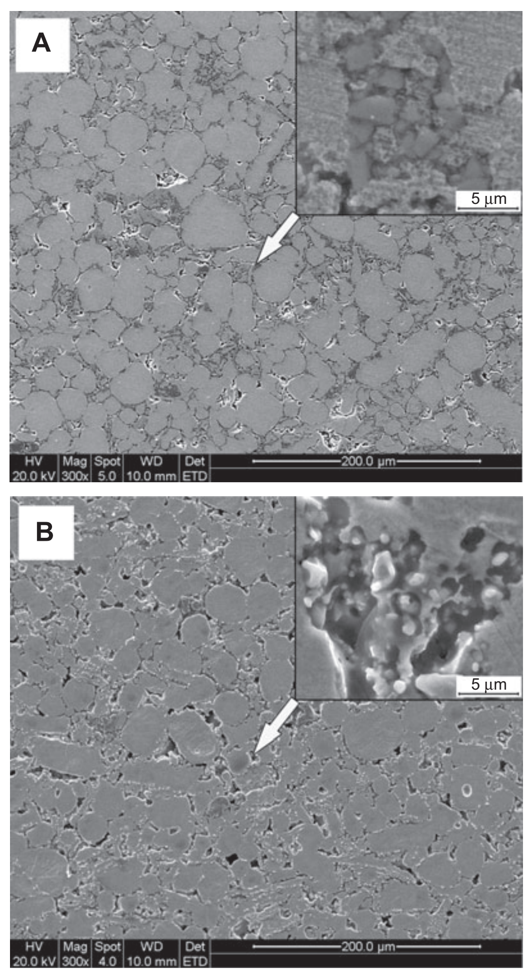

Figure 1 SEM of TiC/316L composites: (A) prepared by conventional PM technique; (B) prepared by WMS.

prepared by WMS exhibited some rounded and isolated pores.

It is important to note that the porosity level in the conventional P/M species was higher when compared to WMS compacts (Figure 1). This correlates with the densification results shown in Table 1. On the other hand, there was a difference of the pore morphology between conventional P/M and WMS species. The conventional P/M species contain mostly irregular, intergranular pores. In contrast, some isolated, intragranular pores are observed in WMS species, and the pores are more rounded. In the case of WMS, owing to its warm compaction process in the initial stage, this treatment is able to improve the plastic deformation capability of 316L stainless steel matrix powders [14], enhancing the densification of green compacts, and to increase the contacts of particles. In reality, there are more contacts on each particle for WMS species, and each contact enlarges and merges to eventually form isolated pores. Moreover, the pores of WMS species are more rounded when compared with that of the conventional P/M species. This can be attributed primarily to the different sintering cycle. It must be pointed out that the WMS species achieved through microwave sintering get heated up rapidly. The faster heating rate results in the enhancing of sintering kinetics during sintering [15], and therefore, it influences neck growth because of faster material transport, contributing to the pore rounding.

Figure 2 shows the X-ray diffractogram (XRD) for the composites prepared by two different technical routes. An XRD pattern of the powder mixture without sintering (Figure 2A) was present for comparison. It can be observed that the constituent phases in the composites are TiC and γ-Fe. No other carbides or intermetallic compounds were identified. Careful examination of Figure 2 reveals that curves of Figure 2B and C exhibited reflection peaks of TiC growing weaker compared to the curve of the powder mixture without sintering, and the position of γ-Fe reflection shifted to a lower angle.

The change of strength of reflection of TiC peaks and the change of position of reflection of γ-Fe peak in Figure 2 indicated the dissolution of TiC and the formation of a solid solution. Furthermore, compared with the XRD of conventional P/M species, the TiC diffraction peak of WMS

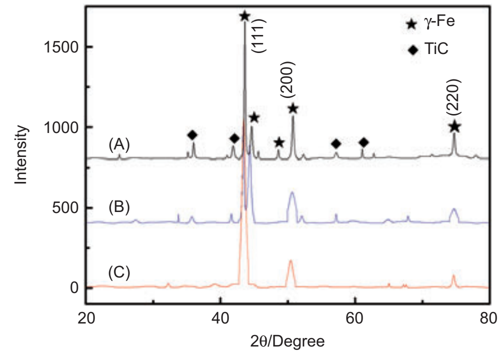

Figure 2 XRD patterns of 5 wt.% TiC/316L composites: (A) powders; (B) prepared by conventional PM technique; (C) prepared by WMS.

species in this study is smaller. Many researchers [15–17] have reported that the application of microwave technology could decrease the activation energy of sintering and increase the sintering kinetics, so the dissolution of TiC and the formation of the solid solution at the WMS process were easier to be observed compared to the conventional P/M process.

3.2 Densification and microhardness measurements

Table 1 presents the relative densities after sintering and hardness of composites prepared by two different preparation methods, respectively. The species obtained by WMS exhibited a higher relative density compared with the conventional P/M species. This clearly suggests that the WMS process resulted in a good densification for the WMS species, as do species processed by the conventional P/M process. This was due to the higher initial density before sintering and the different heating mechanism between vacuum sintering and microwave sintering. Shokrollahi [14] pointed out that the process of warm compaction can result in the increase of plastic deformation capability of 316L stainless steel matrix powders, which had helped to improve the particle rearrangement. As a result, the densification of the green body increases. On the other hand, the heating during conventional sintering occurs due to surface heating, which was a thermal conduction from the

Material	Preparation technique	GD (%)	RD (%)	Hardness (HV)	E_{corr} (V)	E_{pit} (V)	I_{corr} (μA/cm²)
5 wt.% TiC/316L	Conventional PM	66.3	90.6	208.2	-0.27481	-0.07253	0.07991
5 wt.% TiC/316L	WMS	70.2	94.8	332.4	-0.19865	0.08892	0.02910

Table 1 Effect of preparation technique on the densification and electrochemical response of composites.
GD, green density; RD, relative density.

outside to the inside of the species [18]; however, the volumetric heating during microwave sintering was initiated throughout the species [13], which resulted in a higher relative density.

From Table 1, it can be found that the species prepared by WMS exhibited higher hardness. This can be attributed to a better densification during WMS.

3.3 Wear resistance

Figure 3 showed the volume loss of composites. As expected, the species prepared by WMS exhibits significantly better wear resistance, less wear loss than that of the conventional P/M counterpart. This can be explained by Eq. (1), showing the dependence of wear loss to hardness [12]

$$V=\frac{kPL}{H} \tag{1}$$

where V is the volume loss, P is the applied load, L is the sliding distance, H is the hardness of the species, and k is the wear coefficient. Eq. (1) indicates that the volume loss of a species is inversely proportional to the hardness of the material. In this case, the WMS species exhibits higher hardness (Table 1); thus, it experiences the lower volume loss during the sliding wear process.

Figure 4 shows the surface of composites prepared by two different preparation methods after wear tests (sliding distance 1000 m). It is clear from the optical micrographs of the worn surface (Figure 4) that the conventional P/M species showed the severity of material removing, consisting mainly of large, angular, and non-uniformly damaged spots, while the WMS species showed a relatively smooth surface, consisting mostly of continuous longitudinal scratches and grooves. The reasons why the extent of surface damage was much slighter in WMS species compared with that of the conventional P/M species were already addressed for the sufficient sintering of WMS species, which result in a higher densification, and a stronger interface bonding forms between the reinforcement and the matrix. Alpas A.T. et al. [19], F. Velasco et al. [20], and Ashok K.S. et al. [21] reported that a strong bonding between the reinforcing particle and the matrix is desirable for good wear resistance, and so, the material was restrained and removing was prevented. Moreover, the worn surface of the conventional P/M species exhibited a small number of holes, which may be attributed to the removal of the material. No exposed reinforced particles on the worn surface of the WMS species were found (Figure 4B). It also demonstrated that a stronger combination between the reinforcement and the matrix for the WMS species existed, which was advantageous to the improvement of the wear resistance [22]. The main wear mechanism is grooving and abrasive wear for the TiC/316L

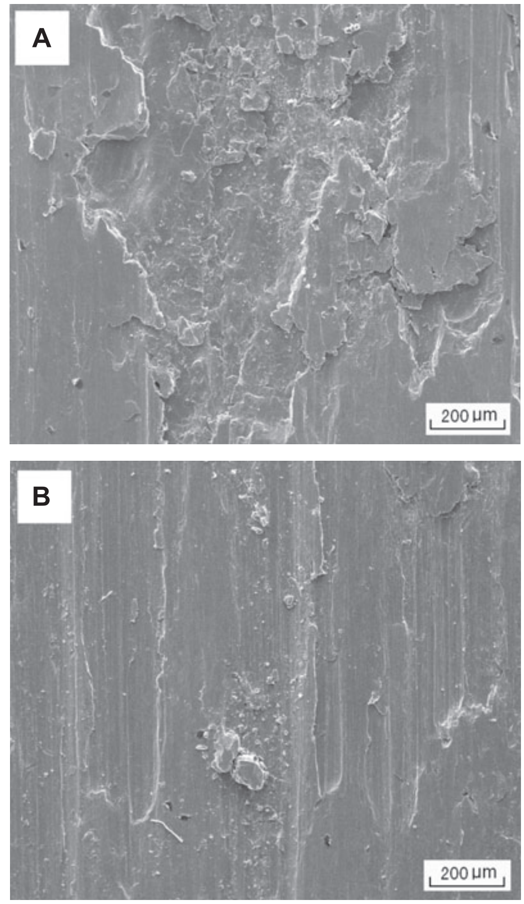

Figure 4 SEM micrographs of the worn surfaces of the TiC/316L composites at 100 N (sliding distance 1000 m): (A) prepared by conventional PM technique; (B) prepared by WMS.

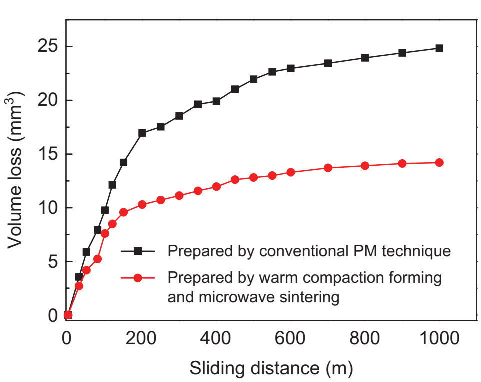

Figure 3 Volume loss vs. sliding distance of TiC/316L composite at 100 N.

stainless steel composites prepared by WMS, whereas it was cutting and abrasive wear for the conventionally P/M composites.

3.4 Corrosion behavior

The effect of the preparation method was also assessed in polarization tests performed in 3.5 wt.% NaCl solution at room temperature. Figure 5 shows the polarization curves for TiC/316L composites prepared by two different technical routes. Their corrosion potential (E_{corr}), corrosion current density (I_{corr}) values (obtained both from Tafel-type fit and linear polarization technique), and E_{pit} values are summarized in Table 1. A closer examination of the corrosion behavior clearly indicates that using WMS shifted the E_{corr} to more noble values and improved the corrosion rate, reducing the I_{corr} in the active region. It is apparent that composites prepared by WMS have a higher potential and a lower current density.

Figure 6 presents the results of the immersion tests as mass loss of the composites with respect to time in 15 wt.% H_2SO_4 at room temperature. For both composites, corrosion progressed rapidly at the early stages and then slowed down. Compared to the WMS species, composites prepared by the conventional P/M exhibited a higher mass loss at extended duration times, especially at comparatively long duration times (i.e., over 2 days).

The differences in the corrosion rate can be attributed to the fact that composites prepared by WMS show a high relative density. A high densification entails closure of interconnected porosity. Consequently, the access of a corrosive medium into the pores is greatly reduced, which in turn reduces the corrosion rate [6, 23].

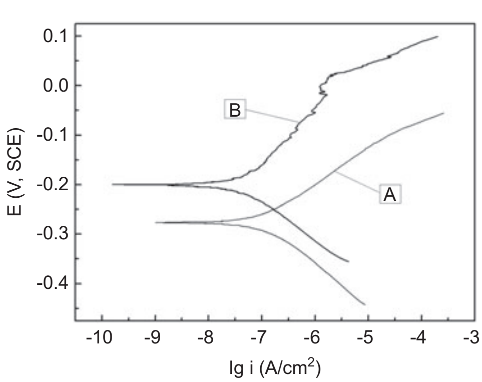

Figure 5 Polarization curves for TiC/316L composites in 3.5 wt.% NaCl environment: (A) prepared by conventional PM technique; (B) prepared by WMS.

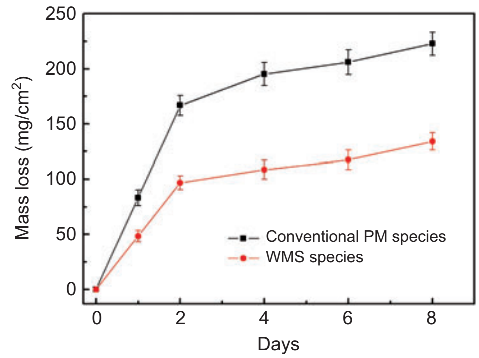

Figure 6 Mass loss of TiC/316L composites as a function of duration time in 15 wt.% H_2SO_4 environment.

Figure 7 shows SEM micrographs of the corroded surface of the examined composites after a duration of 8 days in 15 wt.% H_2SO_4 at room temperature. It is clearly seen that conventional P/M species presents a heavier

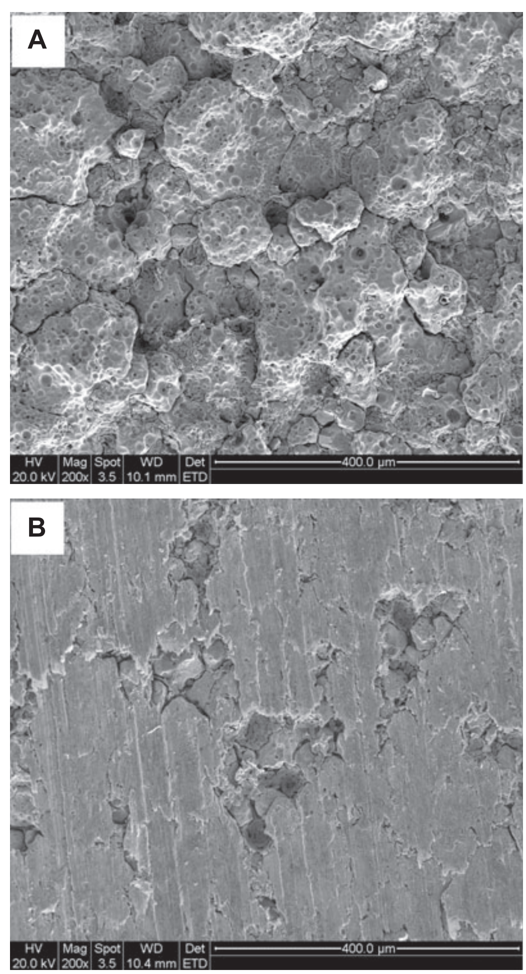

Figure 7 SEM micrographs showing the surface of TiC/316L composites exposed to 15 wt.% H_2SO_4 for 8 days: (A) prepared by conventional PM technique; (B) prepared by WMS.

surface damage compared to WMS species. Moreover, lots of pore spaces were present at the corroded surface of the conventional P/M species, which may be attributed to the removal of material, whereas fewer pores can be seen in the corroded surface of the WMS species. Extensive corrosion pit formation on the surface and through the inner part had taken place for the conventional P/M species.

4 Conclusions

TiC/316L stainless steel composites by combining WMS were successfully fabricated.

The WMS species exhibited significantly superior densification, higher hardness, and better wear resistance when compared with the conventionally P/M processed counterpart.

Compared with the conventional P/M, WMS results in a higher potential and lower current density for TiC/316L composites.

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