Microstructure and Mechanical Properties of 440C–TiC Composite Steels Produced through Powder Metallurgy Processing

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This study sifted the commercial AISI 440C steel powders by using the matrix proposed previously with addition of TiC powder (25, 33 and 40 wt%) to produce a new composite steel with high hardness and strength after powder metallurgy, sintering and heat treatment. The mixing was done by ball milling, sintered at three different temperatures: 1473 K, 1573 K and 1673 K, and followed by a series of heat treatments. The experimental results showed the highest TRS value at 1502.6 MPa, and porosity was decreased to 0.18% after 440C steel was added with 25 wt% TiC powders by sintering at 1673 K. All of the specimens after sintering and conventional heat treatment did not show improvement on the hardness and TRS, instead showed decreases.

KEY WORDS: AISI 440C; TiC powder; powder metallurgy; TRS and porosity.

1. Introduction

Powder Metallurgy (P/M) is a metal processing technique involving production of metal powders and their consolidation into near net shape components. Over the past 10 years, industrial tool steels for cutting or wear of materials have used metal–ceramic composite steels, whereas, powder metallurgy methods offer two very different types of materials with a view to achieving lightweight, high strength, hardness and wear resistance steels.1–4) During last few decades, the demand for lower production costs and the reduction of weight resulted in the use of P/M composite steels. Conventional sintered P/M parts generally have more than 5 vol% porosity. However, enhanced P/M and sintering techniques can be applied to obtain higher densities and improved porosity in the sintered parts.5–6)

Metal matrix composites are known to exhibit exceptional wear resistance; therefore, several investigators used titanium carbide (TiC) as a reinforcing medium in a ferrous metal matrix in order to enhance this property.7–10) TiC has proved its suitability in Fe or Fe base alloys due its high hardness, low density and chemical stability with Fe-based alloys. Pagounis et al. used a similar type of specimen for impact toughness measurements of tool steels, which were reinforced with TiC particles and synthesized by powder metallurgical techniques.11) Most of the work on iron-based composites has involved TiC reinforcement, which is introduced in the iron matrix through P/M route.11)

ELMAX is a high chromium–vanadium–molybdenum alloyed steel with the high wear resistance, high corrosion resistance and very good dimensional stability.12) ELMAX has a similar machining ability, physical properties, chemical composition and heat treatment procedures for AISI 440C grade. The hardness can be achieved HRC 61 (HRA 81.8) after commercially heat treatments. In this study, the ELMAX steel powder was used as the 440C substrate, which was sifted and added with 25, 33 and 40 wt% of TiC powders to explore the effects of three different sintering temperatures and heat treatment methods on the strength and hardness of composite materials.13) It also evaluated the effects of mechanical properties and microstructures on 440C–TiC composite steels after P/M, sintering and heat treatments.

2. Experimental

The chemical composition (mass%) of the 440C steel powders used in this study was as follows: 1.0% C, 0.8% Si, 0.1% Mn, 17.0% Cr, 0.2% Ni, 0.005% P, 0.025% S and 80.87% Fe. Honeywell Microtrac X 100 laser was used to analyze the size of powders. The average particle sizes of 440C steel powders were 9.3 μm and specific gravity was 7.667 g/cm3. Meanwhile, the size of TiC additive was 1–2 μm and specific gravity was 4.9 g/cm3. 440C steel powders were added with different amounts of TiC to prepare for powder metallurgy under three different sintering temperatures.

This experiment used a vacuum sinter furnace with vacuum control between 5.3–6.7×10⁻³ Pa. After sintering, the specimens were processed with quenching (1 373 K) and tempering (793 K for 2.5 h and repeated for 3 times). The heat treatment was performed in a commercial Germanic
Specimens were placed in a vacuum furnace and then heated at 1 373 K for 1 h followed by N₂ cooling to room temperature. Various material characterization techniques were used to evaluate the effects of 440C–TiC composite steel through sintering process and heat treatments. The specimens were tested for apparent porosity, micro-hardness, transverse rupture strength (TRS) test, and SEM inspections.

The SHIMADZU universal material test machine with a maximum load of 25 tons was used for the TRS test and the 3-point bending tests (CNS 3940 G2033). L was 30 mm, b and h were 5 mm in the equation $R_{num}=3FLk/bh^2$, respectively. The specimen dimensions of the 3-point bending test were $5 \times 5 \times 40$ mm. Porosity test followed the ASTM C373-88 standard. Hardness tests were measured by HRA with loading of 60 kg, which complied with the CNS 2114 Z8003 standard.

### 3. Results and Discussion

**Figure 1** represents the SEM micrographs of the original 440C steel powders and TiC additive powders. Figure 1(a) shows the sifting classification of 440C steel powders below 10 μm, the particle sizes were small, uniform and spherical. Figure 1(b) shows the appearance and polygonal shape of TiC powders, and particle size was 1.5 μm. Sintering temperature has obvious effect on porosity of sintered materials. Higher sintering temperature would lead to lower porosity. **Figure 2** shows the porosity of 440C steel added with different amounts of TiC powders after sintering at 1 473 K, 1 573 K and 1 673 K. Under higher sintering temperature, heat activation would increase the atomic diffusion rate to fill the empty holes at longer distance. It showed that the porosity of composite steels can be less than 1% after sintering at 1 673 K. Meanwhile, the porosity would slightly increase with the addition of TiC during the strengthening phase at the same sintering temperature.

Normally, the TiC powders would be added to iron-based materials because of TiC and Fe are immiscible and result in the microstructure of mesh-like distribution. Therefore, an increase in the TiC amount would increase the mesh structures, and hinder the sintered compaction. When these TiC powders increased, the location of the surrounding contacts would be difficult to be filled during sintering. The porosity would increase with addition of TiC at the same sintering temperature. Moreover, the particle sizes of TiC and 440C stainless steel powder were small, which would lead to small pores between powders and particles, so they are easily filled. Atomic do not need reach a long distance to fill the void during sintering process. The sintered composite steels would have the more density and low porosity at high sintering temperature.

The porosity of 440C steel were added with different amounts of TiC powders after sintering at 1 473 K, 1 573 K, and 1 673 K, as well as heat treatments, as shown in Fig. 2. The parameters of heat treatment were: 1 373 K quenching, 793 K tempering 2.5 h, and repeated for 3 times. When the temperature heated to 1 373 K for 1 h, although the heat activation was lower than sintering process, it still was able to densify the sintered materials. The heat energy of interface was large which increases the densification of driving force during heat treatment. It can be found that the porosity was significantly decreased after sintering at 1 473 K and heat treatments. Otherwise, the composite steel would be very dense after sintering at 1 673 K, there was no clear decline after heat treatment.

**Figure 3** shows the hardness and TRS of 440C steel added with different amounts of TiC powder after sintering at 1 473 K, 1 573 K, and 1 673 K. Higher sintering temperature would result in higher density and hardness of the sintered composite steel. It shows that the hardness has a slight increase in the TiC strengthening phase. The maximum hardness was HRA 85.3 (HRC 67.7) which appeared in 440C steel added with 40% TiC after sintering at 1 673 K. It can be found that higher sintering temperature would increase TRS rapidly. The highest TRS obtained in this study was 1502.6 MPa, which appeared in 440C steel added with 25% TiC after sintering at 1 673 K. At the same sintering temperature, increasing the amount of TiC would slightly
increase the porosity, but decrease the TRS of sintered materials. It was observed that the small amount of TiC was enough to improve the structure, hardness, and strength of sintered materials.

Figure 4 shows the hardness and TRS of 440C steel added with different amounts of TiC powder after sintering at 1473 K, 1573 K, and 1673 K, and heat treatments. Comparison with Fig. 3 showed that all the hardness after heat treatment was lower than the sintered result. The hardness of 440C added with 40 wt% TiC was HRA 85.3 (HRC 67.7) after sintering at 1673 K, but decreased to HRA 82.7 (HRC 62.8) after sintering at 1673 K and heat treatment. Especially for sintering at 1473 K followed by heat treatment, the porosity significantly decreased after heat treatment, which in turn improved the hardness. TRS of all composite steels after heat treatment was lower than sintered results. The TRS of 440C added with 25 wt% TiC was 1502.6 MPa after sintering at 1673 K, but decreased to 1290.5 MPa after sintering at 1673 K and heat treatment. Because of the high temperature tempering, carbide of martensite stainless steel tended to precipitate. The content of over-chromium elements would have a large chromium carbide precipitation. Although the precipitation caused strengthening effects, the high tempering temperature resulted in coarse grain size and affected the solid solution strengthening. The hardness and TRS of composite steels were reduced after sintering and heat treatment.

Figure 5 presents the SEM micrographs of 440C steel powders added with 25, 33, and 40 wt% TiC through 1573 K sintering. The microstructures were incomplete after sintering at 1573 K. Many pores were present in the sintered materials which resulted in lower hardness and TRS. Figure 6 represents the SEM micrographs of 440C steel powders added with 25, 33, and 40 wt% TiC through 1673 K sintering. The microstructures were complete and dense after 1673 K sintering. The black particles of TiC were surrounded with grey areas of high chromium steel matrix, as shown in Fig. 6(a). TiC powders produced a continuous mesh structure to improve the strength of sintered material, as show in Figs. 6(b) and 6(c).

Figure 7 shows the SEM micrographs of 440C steel powders added with 25, 33, and 40 wt% TiC through 1673 K sintering and heat treatments. Increasing the addition of TiC powders would increase the mesh structure, and improve the hardness of sintered materials. Figure 8 is the high magnification SEM micrographs and EDS analysis of 440C steel powders added with 25 wt% TiC through 1673 K sintering and heat treatments. Figure 8(a) clearly shows that the round and meshed structure of TiC particle is continuous and surrounded with the martensite substrate. Moreover, the microstructure appeared with tempering martensite after 1373 K quenching and 793 K tempering, as shown in Fig. 8(b). It can be found that many of smaller and uniform carbides precipitated in the substrate. These particle sizes of carbides were near 1 μm. From the EDS analysis as shown in Fig. 8(c), the Ti and Cr elements were
transferred to the martensite structure and present in the matrix.

Figure 9 shows the mapping analysis of 440C steel powders added with 25 wt% TiC through 1673 K sintering. According to the distribution of Fe and Ti elements, it can be confirm that the black particles are TiC powders. The surrounded grey area was high-chromium steel substrate. Because of the liquid phase sintering process, Ti elements can spread to the gap between the particles with the chromium elements, and be evenly distributed within the material. However, increasing the addition of TiC would enhance the density of mesh structure. Figure 10 shows the fracture surfaces of the 440C steel added with different amounts of TiC powders through 1673 K sintering. Figure 10(a) shows that the dimpled rupture is present in the sintered material. Figures 10(b) and 10(c) present the brittle fracture microstructure after TRS tests in the brittle intergranular failure mode. Increasing the addition of TiC powders would increase the porosity and enhance the chance of brittle fractured, it was easy to achieve the brittle property of the sin-
tered materials.

4. Conclusions

(1) AISI 440C steel powders were added with different amounts of TiC powder through powder metallurgy and heat treatments to produce new composite steels. Experimental results showed that the optimal P/M and sintering processes were adding 25 wt% TiC powder to 440C steels through 1 673 K sintering.

(2) The microstructures were complete and dense after 1 673 K sintering. TRS reached 1502.6 MPa, porosity decreased to 0.18%, and hardness increased to HRA 85.3 (HRC 67.7). But TRS decreased to 1290.5 MPa, hardness reduced to HRA 79.4, and porosity increased to 0.34% of 440C–TiC composite steels after 1 673 K sintering and heat treatments. These results showed that the sintered steels have higher strength and hardness after processed with the method proposed in this study.

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