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The Effect of Milling Time on Microstructure and Wear Behaviours of AISI 304 Stainless Steel Produced by Powder Metallurgy

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In this study, the effect of milling time on wear behavior of the AISI 304 stainless steel produced by the mechanical alloying method was investigated. In the study element powders were prepared and mechanically milled at five different milling times (30, 60, 90, 120, and 150 min) in a mechanical alloying device. The milled powders were pre-shaped and sintered at 1300 °C for 1 h and cooled to room temperature in the furnace. Produced samples microstructure and XRD examination were carried out. Wear tests were performed using a pin-on-disc type wear testing device. In this study, the hardness values were found to have increased by increasing milling time. The maximum hardness values were measured for the 150 min milled samples. The lowest weight losses were measured with 150 min milled samples. The wear test results were compatible with hardness results.

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PACS/topics: AISI 304, mechanical alloying, milling time, hardness, wear

1. Introduction

Austenitic stainless steels are the most commonly used type of stainless steel. This is due to their high corrosion resistance in many corrosive environments and their ability to maintain this property even at relatively higher temperatures [1, 2]. While austenitic stainless steels may be subjected to work hardening under tension, it is not possible to harden such stainless steels using heat treatment methods due to their austenitic microstructure [1]. Their lower wear resistance, compared to other stainless steels, limits usage areas of this group of steels [3, 4]. The most significant advantages of producing austenitic steels by powder metallurgy include relatively lower material and energy use, and the ability to produce almost all materials in desired composition and size [4, 5]. The ability to manufacture a product in the final dimensions eliminates the costs associated with other processes. To increase the hardness of austenitic stainless steel without any additional process costs using powder metallurgy is possible by microstructure control. It is possible to control the microstructure of austenitic steels by controlling the powder size, milling time, sintering temperature, and sintering duration. Hence, this study investigates the effects of the mechanical alloying time on the microstructure hardness and wear performance of AISI 304 austenitic stainless steels.

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2. Experimental procedure

Elemental powders were used in the mechanical alloying (MA) of AISI 304 austenitic stainless steels. Table I shows the chemical composition of the AISI 304 alloy produced in the experimental studies.

TABLE I

The chemical composition of the AISI 304 austenitic stainless steel

Element	Cr	Ni	Mn	Si	C	Fe
wt%	18	8	2	0.8	0.08	Bal.

Together with iron powder with 99.9+% purity and < 10 μm grain size, Cr (-325 mesh), Ni (3–7 μm), Mn (-325 mesh), Si (1–20 μm), and C (10–20 μm) were used in experimental studies. The elementary powders were mechanically alloyed in a planetary mill using five different alloying times (30, 60, 90, 120, and 150 min). Stainless steel balls 8 mm in diameter, 10:1 ball/powder ratio, and 1% ethanol as process control chemical were used to prevent agglomeration during MA processes. The mill was paused for 10 min after each 15 min MA interval in order to prevent powders from overheating. The size of MA'ed powders was measured with the Malvern brand laser particle sizer. The MA'ed stainless steel powders were cold-pressed (620 MPa) to ø12 × 6 mm green compacts. The green compacts were sintered using 4 °C/min heating rate at 1300 °C in nitrogen + 5H% environment for 60 min. The samples were prepared for microstructure examinations using standard metallographic processes, and electrolytic etch with 10% oxalic acid for 15 s. After etching, the samples were characterized with scanning electron microscopy + energy dispersive spectroscopy (SEM+EDS) examinations, X-ray diffraction (XRD), and hardness and density measurements. Wear tests were performed using a pin-on disc-type wear testing device at room temperature and with 1 ms^{-1} sliding speed in accordance with ASTM:G99-5 standards. Three different loads (15, 30, and 45 N) and five different sliding speeds were used in wear tests.

3. Results and discussion

3.1. Microstructural characterization

Figure 1 shows powder size, hardness, and density changes in the AISI 304 austenitic stainless steels produced by MA. The powder size analysis given in Fig. 1a shows that the increase in MA time led to a decrease in powder size. The powder-ball-mold wall collision during the MA process leads to cold welding, deformation hardening, and fracturing [6]. Among the austenitic stainless steel powders mechanically alloyed using different alloying times, the lowest average powder size, 22 μm, was measured for the powders alloyed for 150 min.

Figure 1b shows that the hardness value of the material produced from powders increased with increasing powder milling time. This is due to the deformation hardening acting on the MA'ed alloy powders. Figure 2 shows the microstructure SEM images of the AISI 304 austenitic stainless steels produced by MA using five different MA times. As shown by the SEM images, the increase in MA time led to a microstructure with finer grains.

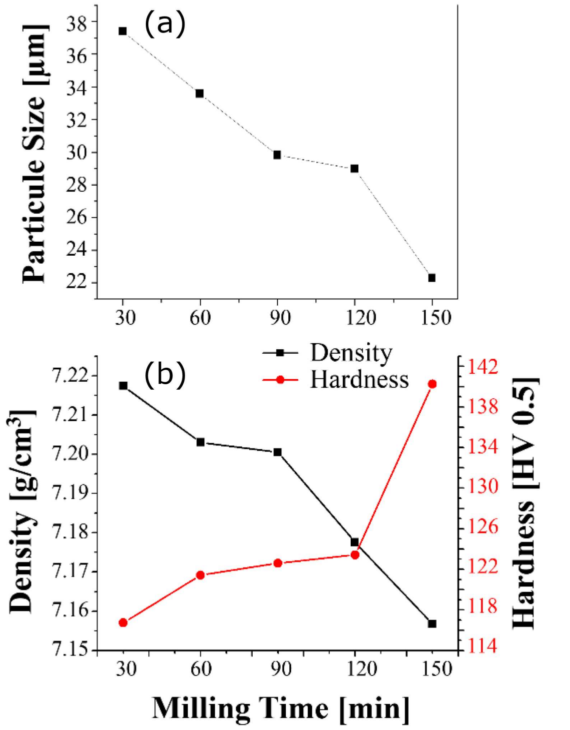

Fig. 1. Powder size analysis (a) and hardness and density changes (b) in the AISI 304 austenitic stainless steels depending on MA time.

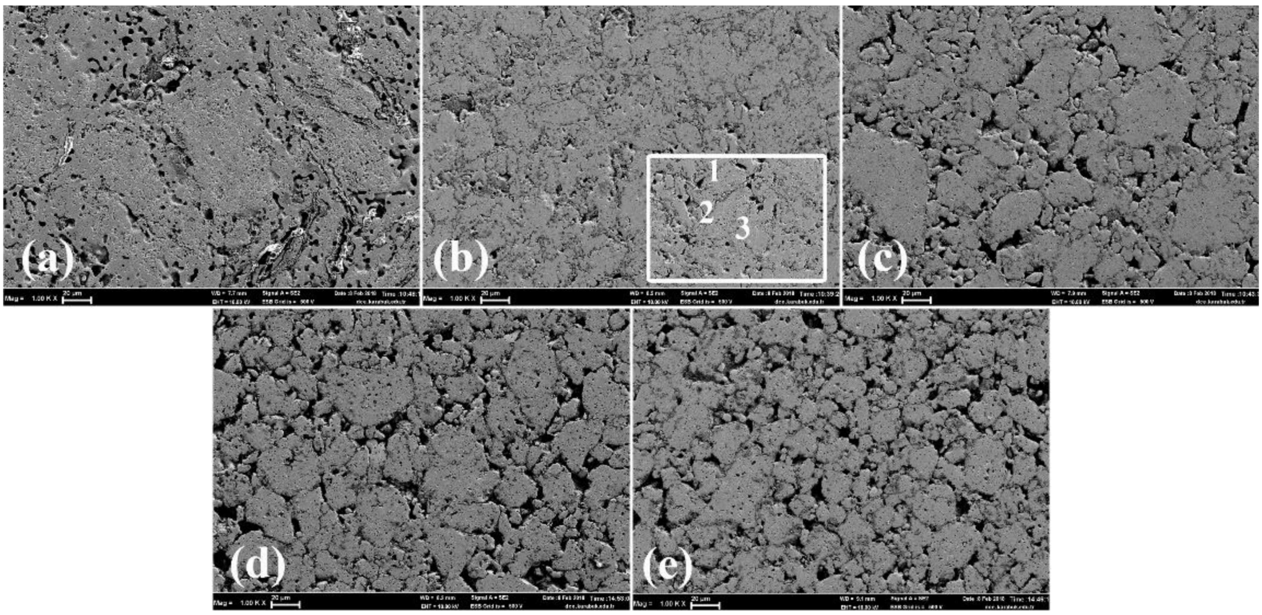

Fig. 2. The SEM images of the AISI 304 austenitic stainless steels produced using five different MA times: 30 min (a), 60 min (b), 90 min (c), 120 min (a), 150 min (e).

Figure 2e shows that 150 min MA'ed alloy has the finest grains compared to Fig. 2a–d. The decrease in the size of grains in the microstructure leads to an increase in material hardness (Fig. 1b). This can be explained by the Hall–Petch equation [7]. The microstructure SEM images shown in Fig. 2 also show that the AISI 304 austenitic stainless steel consisted of porous structures. The powder forming process was performed using a rigid die compaction, which is the most preferred mold type due to its low cost. This causes porosity in the structure. It can be observed that the size of the pores in the microstructure decreased with increasing MA time. A former study reports that the MA time is an effective process parameter for the powder size of the alloy in the MA process [8]. The decrease in the size of powder grains leads to a decrease in the pore size. Figure 1b shows that the material density decreased in spite of the decrease in the pore size. Increasing MA time leads to an increase in the deformation hardening in powders. The increase in the powder hardness decreases the compressibility of the powder, which leads to a decrease in part density. Table II shows the EDS analysis results for the austenitic stainless steel sample MA'ed for 60 min. It can be easily observed in the EDS results that some oxidation occurred in zone (1). The performance of MA processes in a non-atmosphere-controlled environment may cause some oxidation in the sample. Figure 3 shows the XRD results of the AISI 304 austenitic stainless steels produced by MA.

The XRD results shown in Fig. 3 shows that γ-austenite, α'-martensite, and Fe_3O_4 phases formed in the structure of the alloys.

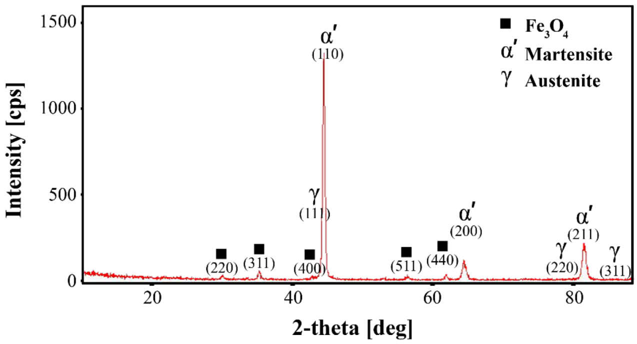

Fig. 3. The XRD results of the AISI 304 austenitic stainless steels produced by MA.

TABLE II

The results of the EDS analysis (elements contents in wt%)

Zone	Fe	Cr	Ni	Mn	Si	C	O
1	3.47	40.80	0.52	17.91	0.38	1.58	35.33
2	74.95	11.51	9.63	1.59	0.15	2.16	–
3	1.56	60.73	0.57	30.88	3.21	3.05	–

3.2. Wear test results

Figure 4 shows weight losses and wear rates obtained as a result of the wear tests performed for the AISI 304 austenitic stainless steels produced by MA using five different MA times.

Figure 4 shows that the 30 min MA'ed alloy had the highest weight loss in wear tests performed under 15 N (Fig. 4a), 30 N (Fig. 4b), and 45 N (Fig. 4c) load. As shown by the charts, the increase in the load applied during the test led to a decrease in the weight loss after 1200 m. The alloys MA'ed for 150 min had the lowest weight loss under all loads tested. Considering the hardness values, the weight loss of the material decreased with increasing hardness and increased with increasing load. The increase in alloying time led to a decrease in the grain size, which led to a decrease in the hardness of the alloy.

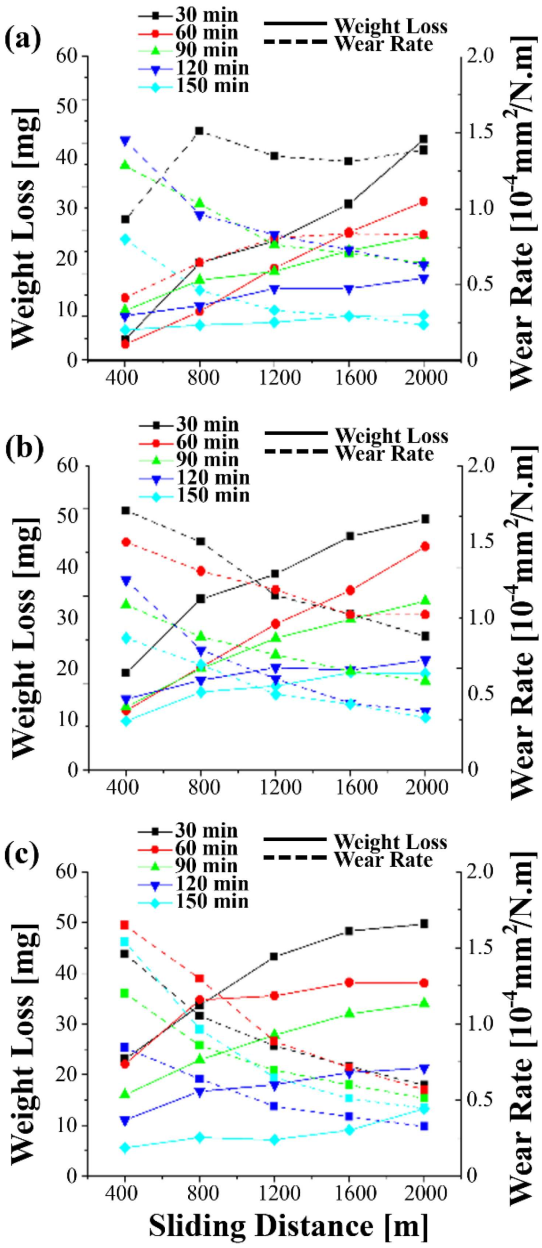

Fig. 4. The weight losses and wear rates obtained as a result of the wear tests performed for the AISI 304 austenitic stainless steels produced by MA using five different MA times: 15 N (a), 30 N (b), 45 N (c).

4. Conclusion

The results of the present study which investigates microstructures and wear behaviors of AISI 304 austenitic stainless steel alloys produced by the mechanical alloying method using 5 different mechanical alloying times are given below. The AISI 304 stainless steel alloys were produced using the MA method for different alloying times. The powder size analysis showed that there was a decrease in the powder grain size with increasing alloying time. The increase in MA time led to an increase in the hardness value, and the highest hardness value was obtained for the AISI 304 alloy MA'ed for 150 min. All microstructures were porous, and the density measurements showed that the density decreased with increasing MA time. In line with the hardness results, the wear test results showed that the AISI 304 stainless steel MA'ed for 150 min had the lowest weight loss and wear rate.

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