

AUSTENITE-MARTENSITE TRANSFORMATION IN NANOSTRUCTURED AISI316L STAINLESS STEEL POWDER INDUCED DURING MECHANICAL MILLING

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Abstract

Mechanical milling as a kind of severe plastic deformation process is extensively used for producing nanocrystalline powder. In this research, morphology, microstructure, phase composition, and hardness changes of AISI316L stainless steel chips during ball milling were investigated. During ball milling, austenite in as-received chips, was transformed to martensite with a grain size of about 10nm leading to an increase in hardness value from about 300HV for as-received chips to 800HV for ball milled sample. Heat treatment was done at two different temperatures to investigate the martensite-austenite transition upon annealing. Martensite induced during ball milling was transformed to austenite by annealing at 700°C partially and completely at 900°C. In the case of annealing at 900°C, the austenite grain size increased with cooling rate.

Keywords and phrases: austenite martensite transformation.

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1. Introduction

Nanocrystalline materials, known as materials with a grain size smaller than 100nm, have attracted attention of scientists due to their different and interesting properties as compared to their coarse counterparts [2, 3, 5]. Some of these properties include: high resistivity, ultra-magnetic behaviour, high thermal expansion coefficient, and high strength [6].

Cold working has been introduced as a top-down approach for producing nanostructures. In mechanical alloying (MA) process, the repeated impact of milling balls causes severe plastic deformation of particles readily producing nano sized microstructure.

During MA process, materials could show three different behaviours depending on their physical and mechanical properties are as follows:

(1) Ductile-ductile system: In this case, ductile components are flattened to platelet shapes by a microforging process in early stages of MA. Then, due to the balls impacts and particles contacts, they are cold welded together. Lamellar structure and increase in particles size can be seen in this stage. With further milling, powder particles are work hardened resulted in increasing of the brittleness; therefore, the tendency for fragmentation is increased. As a result, the powder particles are reduced in size at longer milling time. When the cold welding and fragmentation rates become balanced, particles size is not changed further.

(2) Ductile-brittle system: In this case, ductile metal powder particles are flattened by the ball-powder-ball impacts and brittle particles are trapped in them in early stage of MA. With further milling, the ductile powder particles are work hardened and distribution of brittle particles into the ductile phase becomes more uniform. At longer milling time, alloying will occur only if brittle phase is soluble in ductile phase.

(3) Brittle-brittle system: In this case, only particles size decreases with increasing milling time and alloying does not occur in this system at very low temperatures [4, 10].

Austenite stainless steels are often categorized in two types according to the stability of austenite phase: stable and metastable. Cold working induced by ball milling causes the metastable austenite phase to transform to martensite phase, but it has no effect on the stable austenite phase [7]. Spencer [8] showed that the rate of formation of strain-induced martensite is influenced by existing dislocation structure and can be accelerated, if the initial dislocation density is sufficiently high. In milling process, the high strain rate (about 10^3 - 10^4 s⁻¹) leads the shear bands to be formed. Also, low stacking fault energy (SFE) enhances the work hardening rate [9]. The temperature dependence of the SFE may play the key role in the suppression of the martensite transformation at high strain rates [11].

In the present work, chips of 316L austenitic steel were used as starting material and ball milling technique was used in order to refine the grain size down to nano scale size. Strain induced martensite phenomenon and the effect of heat treatment on phase composition were discussed.

2. Experimental Methods

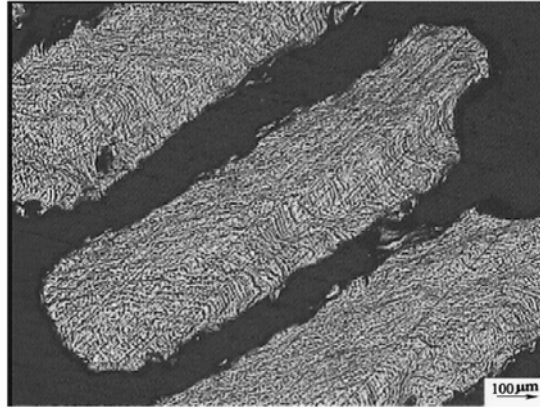
Chips of 316L austenitic steel with composition of (C: < 0.03, Si:1, S: < 0.03, P: < 0.4, Mn: 2, Ni:11, Cr:18, Mo: 2 balance Fe in wt%) were used as starting material. The as-received chips were cleaned by acetone before ball milling. Laboratory planetary ball mill (under Ar atmosphere) was used for ball milling experiments. Milling media consisted of four 20mm diameter balls located in a 120ml volume bowl. The bowl and ball materials were hardened chromium steel. In all ball milling runs, the ball-to-powder weight ratio was about 10:1 and the angular velocity of the bowl and the supporting disc was approximately 400rpm. The hardness of powder particles was also determined by microhardness measurements by using a Vickers indenter at a load of 10g and dwell time of 10s. Three indentations were made on cross-section of each particle to obtain an average value of microhardness. The particles morphology and microstructure

were investigated by optical and scanning electron microscopy (SEM) in a Philips XL30 at an acceleration voltage of 20kV. Structural changes of samples were studied by X-ray diffraction (XRD) in a Philips X'PERT MPD diffractometer by using filtered $\text{CuK}\alpha$ radiation ($\lambda = 0.1542\text{nm}$). Isothermal annealing was carried out to study the thermal behaviour of powder particles. Samples were sealed and then annealed at 900°C and 700°C for 1h in a conventional tube furnace under Ar atmosphere. The structural transitions occurring during annealing were determined by XRD.

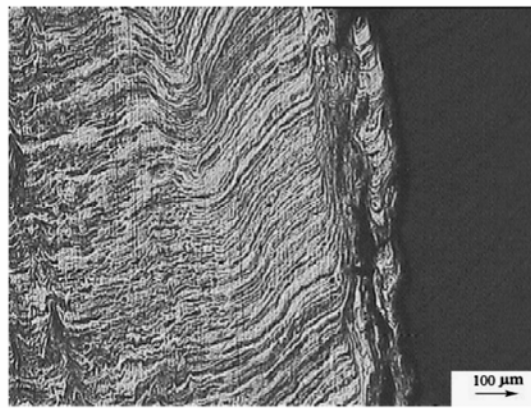
3. Results and Discussion

Figure 1 presents the non-etched morphology of AISI316L at different ball milling times. As can be seen, particle size decreases by increasing the milling time. Also, spherical and laminar shape of particles can be seen after 5 hours ball milling. It is also obvious in SEM image of 60h ball milled sample (Figure 2).

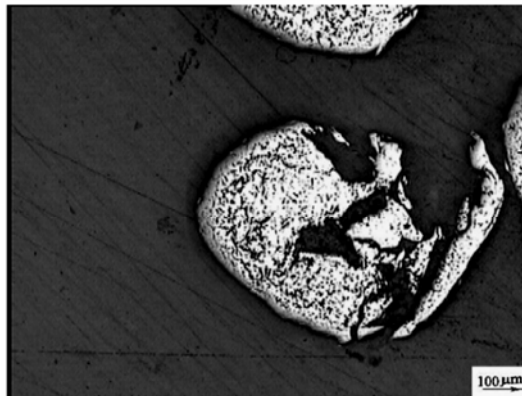
Morphology changing shows that during mechanical milling, lamellar structure in powder particles is obtained. Spherical shape of particles is due to the forge-like operation, which occurred upon them by balls during the process. Impacts of balls cause the particles to be broadened in early times of milling. Pursuing the process, particles become work hardened and hence brittle. During ball milling, the powder particles are repeatedly flattened, cold welded, fractured, and rewelded. Whenever two balls of the milling media collide, some AISI316L powder is trapped between them. The force of the impact plastically deforms the powder particles leading to work hardening and fracture. Due to the high amount of work hardening rate of AISI316L, increasing particle size with milling time was not seen. It also indicates that work hardening rate is more effective than the tendency of particles to be rewelded.



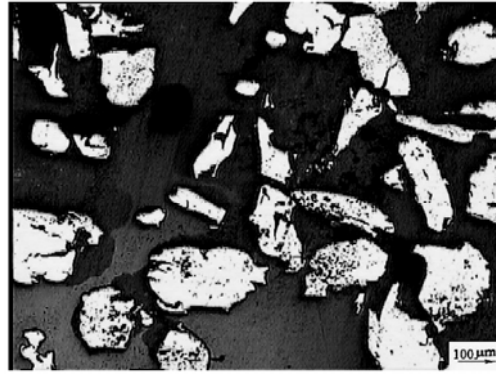
1 hour



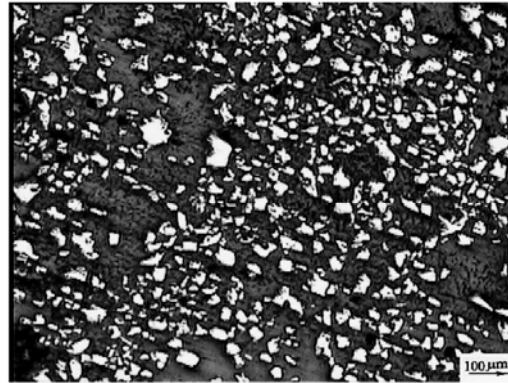
2 hours



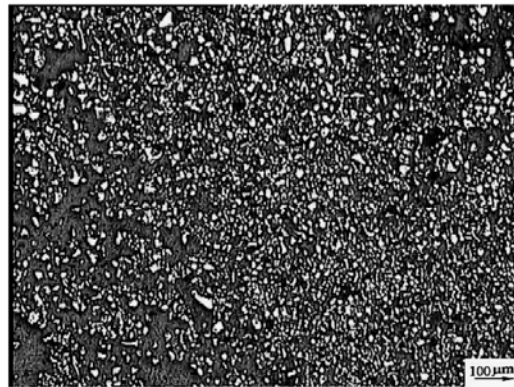
5 hours



15 hours



40 hours



60 hours

Figure 1. Non-etched morphology of AISI316L stainless steel after different ball mill times.

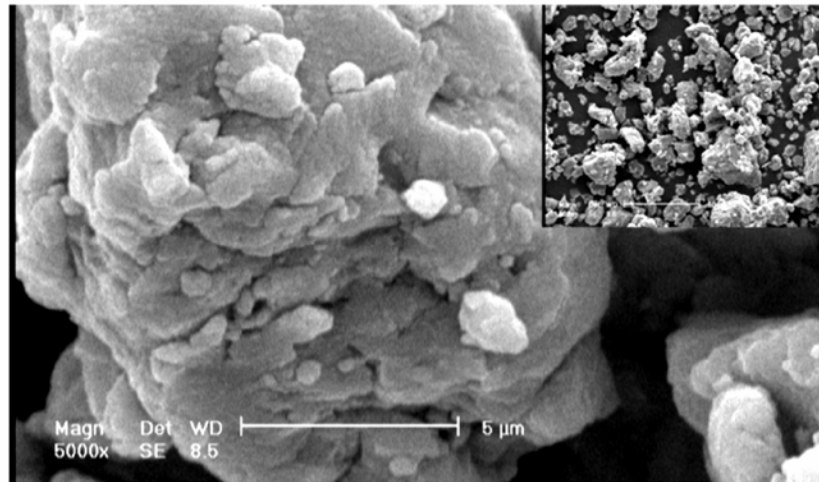


Figure 2. SEM image of AISI316L stainless steel after 60h ball milling.

Hardness changing is shown with a curve in Figure 3. Because the particle size of 60h and 80h samples was smaller than the Vickers indenter, the micro hardness of these samples could not be determined. Because of the formation of martensite phase and increasing dislocations density due to the cold working, the micro hardness of samples increases by milling time. Also, it can be seen that in higher milling time, the slope of curve decreases. This phenomenon can be explained by the following two reasons:

(1) Recovery of dislocations, which occurs due to heat increasing in milling media while milling, is processed, showing the effect of temperature and grain size on stacking fault energy (SFE).

(2) Glutting of dislocations that could happen in higher milling times. As a result, dislocation density does not increase with milling time and particles hardness does not increase either.

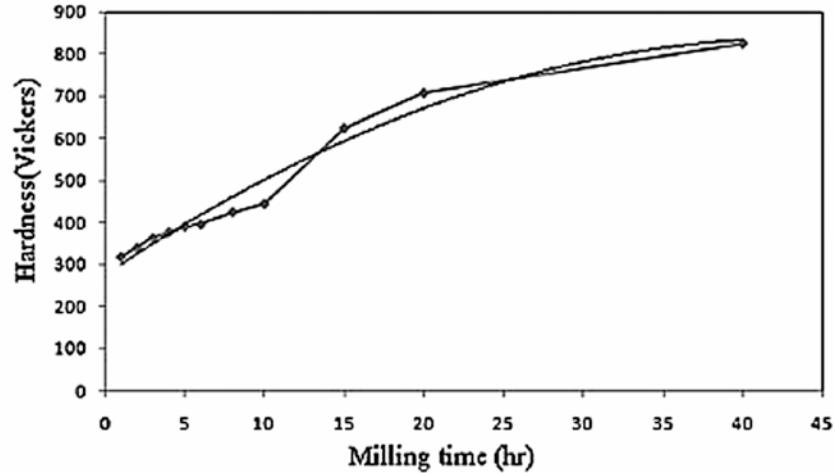


Figure 3. Hardness changes during ball milling.

The XRD traces presented in Figure 4, show the microstructural changes during ball milling process. Only austenite phase is observed in the as-received chips (Figure 4(a)). As can be seen in Figure 4(b), after 20h mechanical milling, the diffraction peaks intensity of austenite is decreased significantly, and instead, the diffraction peaks of martensite are introduced indicating remarkable formation of martensite phase during ball milling process. Increasing milling time to 60h and 80h leads to an increase in fraction of martensite phase. According to Figure 4(d), the austenite is not completely transformed to martensite phase even after 80h milling. In fact, the structure of the metastable austenite is transformed to martensite phase due to the cold working, but the stable austenitic stainless steel is seen to still exist even after 80h ball milling. Also, in Figure 4, broadening of the crystalline diffraction peaks by increasing the milling time as a result of refinement of crystallite size can be seen. The crystallite size and internal strain of austenite and martensite were calculated by analyzing the X-ray diffraction peak broadening. The equation of Williamson and Hall was used to consider the two effects of crystallite size and strain

$$\beta \cos \theta = \frac{K\lambda}{D} + 2A\varepsilon \sin \theta, \quad (1)$$

where β is the diffraction peak width at half-maximum intensity, θ is the Bragg diffraction angle, K is the Scherrer constant (0.91), λ is the wavelength of the radiation used, D is the crystallite size, ε is the average internal strain, and A is a coefficient, which depends on the distribution of strain and it is close to unity for dislocations. The average grain size of the austenite and martensite after 80h was about 10nm. Besides, the average internal strain in the austenite and martensite phases was about 1%.

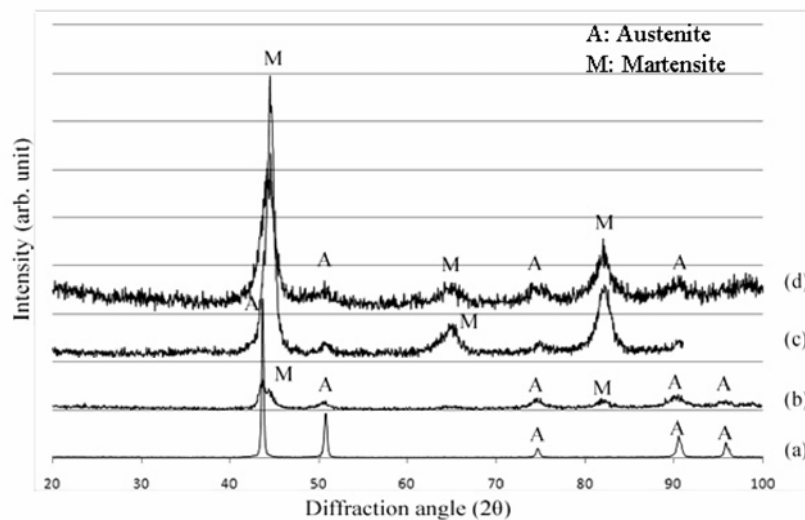


Figure 4. XRD traces of (a) as-received chips and ball milled powders for (b) 20h, (c) 60h, and (d) 80h.

The phase structure of 60h ball milled followed by 1h in 700°C annealed sample, is compared to non-heat treated sample and as received chips are presented in Figure 5.

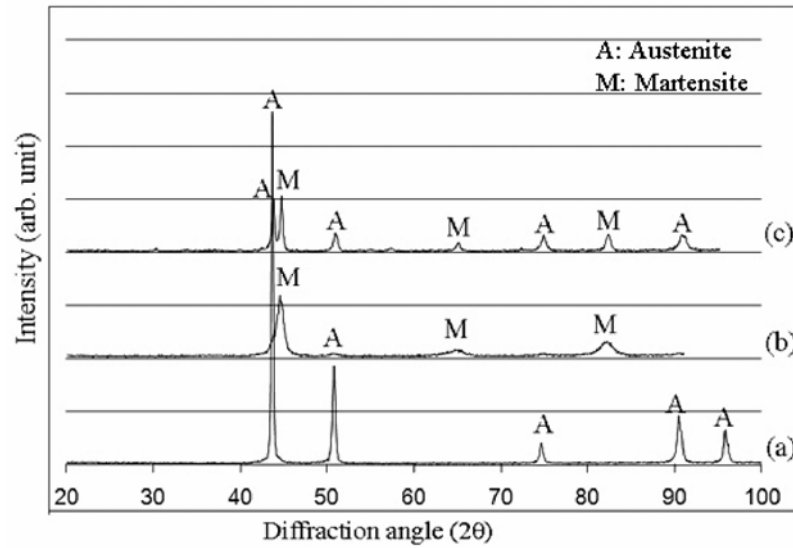


Figure 5. XRD traces of (a) as-received chips, (b) 60h ball milled, (c) heat treated AISI316L stainless steel of 60h ball milled (1h in 700°C furnace cooled).

Increasing of austenite fraction as a result of intensifying the austenite peak intensity can be seen in Figure 5(c) as compared with Figure 5(b). Furthermore, peak width is decreased due to grain growth. In fact, in this condition, martensite to austenite transformation and grain growth due to the annealing takes place. As also shown by Enayati and Bafandeh [1], in this situation, some fractions of martensite phase are transformed to the metastable austenite.

Figure 6 shows the result of severer heat treatments. Heating at 900°C for 1h and cooling in two different conditions, air and furnace have taken place in the 80h ball milled sample. As can be seen, in both cooling conditions, martensite phase is completely transformed to austenite phase, but unexpectedly the grain size of the air cooled sample was broader than furnace cooled (Table 1). In the case of sample which was cooled in furnace, the presence of dislocations prevents the grains to

grow, but for air cooled sample, the effect of cooling intensity overcomes this and as a result, the grain size becomes larger. In fact, the tendency of sample to reduce the grain boundaries energy by grain growth and dislocations energy are competing. In air cooled sample, the cooling intensity could help the grain boundary energy overcome the dislocations energy.

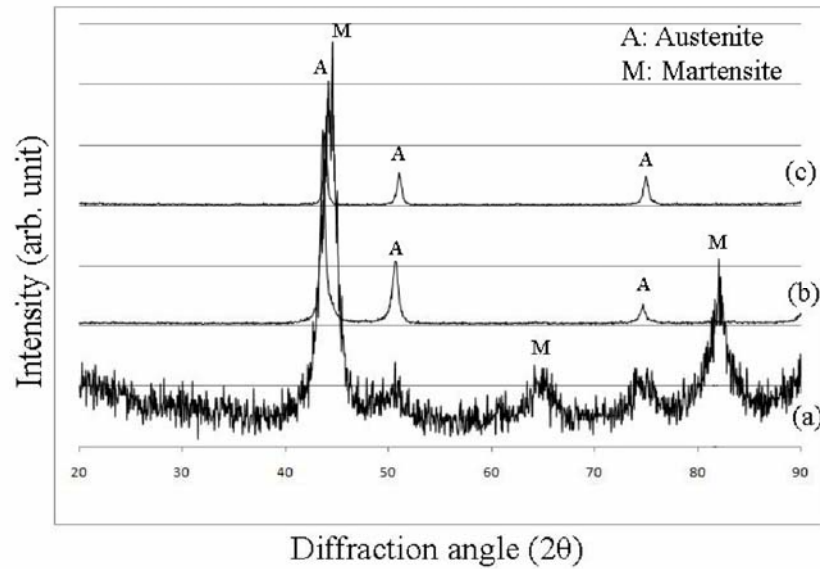


Figure 6. XRD traces of (a) 80h ball milled, (b) 80h ball milled and 1h in 900°C furnace cooled, and (c) 80h ball milled and 1h in 900°C air cooled.

Table 1. Grain size of AISI316L stainless steel powder in different cooling conditions after annealing at 900°C for 1h

Powder air cooled	$D_A = 25.7\text{nm}$
Powder furnace cooled	$D_A = 14.45\text{nm}$

4. Conclusion

Behaviour of austenitic stainless steel chips during mechanical milling was studied. During ball milling, part of austenite in as-received chips was transformed to martensite with a nano scale structure due to the stress induced martensite phenomenon. Hardness value of powder particles after 40h ball milling was twice greater than that of the original chips. The stress-induced martensite was not completely transformed to the austenite during annealing at 700°C. Complete transformation of martensite to austenite happened during annealing at 900°C. These results show that approaching nanostructure from the scrap chips by using ball milling technique can be readily possible and also the formed nanostructure has appropriate thermal stability in high temperatures.

References

- [1] M. H. Enayati and M. R. Bafandeh, Ball milling of stainless steel scrap chips to produce nanocrystalline powder, *J. Mater. Sci.* 42 (2007), 2844-2848.
- [2] H. Hahn and K. A. Padmanabhan, *Advanced Materials and Processing*, Korean Institute of Metals and Materials, Kyongju, Korea, 3 (1995), 2119-2132.
- [3] H. Hahn and K. A. Padmanabhan, *Synthesis and Processing of Nanocrystalline Powder*, Minerals, Metals and Materials Society, Warrendale, PA, (1996), 21-29.
- [4] B. S. Murty and S. Ranganathan, Novel materials synthesis by mechanical alloying/milling, *International Materials Reviews* 43(3) (1998), 101-141.
- [5] A. Nakahira and K. Niihara, *Nanophase and Nanocomposite Materials*, MRS Symposium, Pittsburgh, PA, (1993), 405-418.
- [6] K. A. Padmanabhan, Mechanical properties of nanostructured materials, *Materials Science and Engineering A* 304-306 (2001), 200-205.
- [7] W. F. Smith, *Structure and Properties of Engineering Alloys*, McGraw-Hill, New York, (1969), 300.
- [8] K. Spencer, Strengthening via the formation of strain-induced martensite in stainless steels, *Materials Science and Engineering A* 387-389 (2004), 873-881.
- [9] M. G. Stout and S. S. Hecker, Effects of strain state and strain rate on deformation-induced transformation in 304 stainless steel, Part I, Magnetic Measurements and Mechanical Behaviour, *Metallurgical Transactions A* 13(4) (1982), 619-626.

- [10] C. Suryanarayana, Mechanical alloying and milling, *Progress in Materials Science* 46(1-2) (2001), 1-184.
- [11] J. Talonen, Formation of shear bands and strain-induced martensite during plastic deformation of metastable austenitic stainless steels, *Acta Materialia* 55 (2007), 6108-6118.

