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Formation of a nanocrystalline surface layer on steels by air blast shot peening

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Abstract A nanostructured surface layer was fabricated on two kinds of steels by means of air blast shot peening. The nanolayer shows a sharp boundary to the underlying work-hardened area and good thermal stability up to 873 K. It has much higher hardness than the work hardened region in both the as-treated and annealed states. When using small shot sizes, the nano area can be formed in very short treatment times, and the thickness and continuity of the nanolayer is enhanced. On the contrary, the nanocrystalline region is more difficult to synthesize when using large shot particles, even though the deformed area is much thicker. The effect of particle diameter is attributed to the different collision time and different strain rate of the treated materials.

Introduction

Various severe deformation methods have been developed to fabricate nanocrystalline structured materials, such as ball milling [1–3], high pressure torsion [4, 5] and equal channel angular pressing [6, 7], which have been successfully used to many materials including pure metals, alloys and intermetallics [8, 9]. Nevertheless, there are some difficulties in producing bulk nanostructured materials, for example, contamination and void are hardly avoidable in ball milling [10], while other methods are restrained by the capability of equipment to make larger samples [11].

On the other hand, the surface properties have strong influence on the whole performance of materials, usually the crack initiation and corrosion occurs on the surface with priority, while nanostructured material have showed some attractive novel properties, such as higher corrosion resistance and wear resistance. So it is naturally expected that the global performance can be enhanced when forming a nanocrystalline layer with superior properties on material surface [12–15]. By means of surface treatment based on severe plastic deformation, when the nanolayer is synthesized, it can keep same chemical composition to matrix then without bonding problem compared to heterogeneous surface layer.

In present study, the fabrication of nanostructured surface layer on two kinds of steels by air blast shot peening was carried out, the structure and property of surface layer was characterized by FE-SEM, TEM and microhardness testing. The annealing behavior of nanolayer was also investigated. The effect of processing condition on the character of nanolayer will be discussed.

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Experimental procedure

Two materials were used in present study, one is the silicon steel Fe-3.29Si (Fe-3.29Si-0.01Mn in mass%), The other is one ultra low carbon steel (Fe-0.03C-1.37Mn-0.12Cr in mass%).

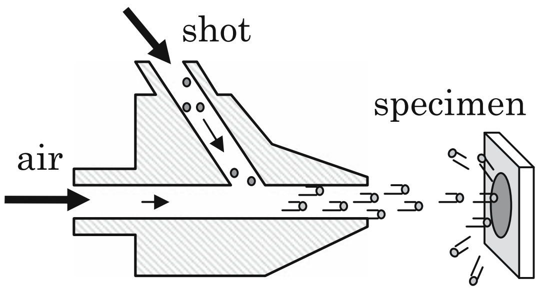


Fig. 1 The schematic illustration of air blast shot peening

The process of air blast shot peening is schematic illustrated in Fig. 1. The processing parameter is shown in Table 1. The 100% coverage corresponds to processing duration of 1 s.

To investigate the thermal stability of nanocrystallite, the shot peened sample was annealed at 873 K for 3.6 ks under the protection of pure Ar by being sealed in quartz tube. The microstructure observation was performed on FE-SEM (JEOL JSM6500) and TEM (Hitachi 800), while the sample were etched by 5% Nital. Microhardness was determined using MVG-G1 Vicker's hardness tester with an applied load 0.98 N for 10 s.

Results

Nanocrystallization in Fe-3.29Si

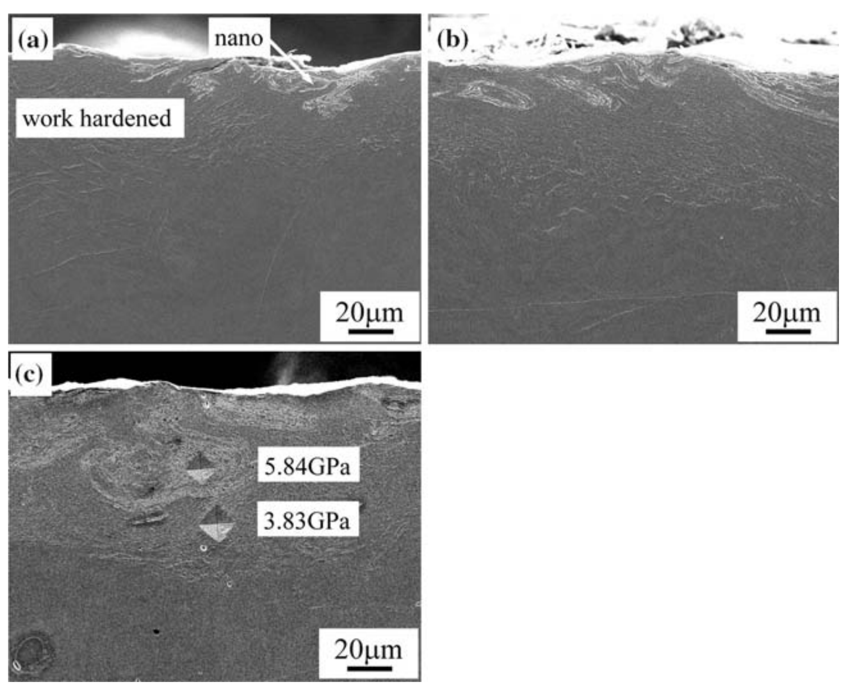
Figure 2 shows the microstructure of shot peened Fe-3.29Si with coverage 3,000%, 10,000% and 30,000%. On the top surface of the samples, there lie some bright regions, which are composed of nanometer-scaled grains confirmed by TEM, as shown in Fig. 3. Beneath the nanocrystallline area, the work hardening area can be seen, while these two regions are separated by sharp boundary. The microhardness of nano region in the as-shot sample with coverage 3,000% attain 5.8 GPa, which is about 1.5 times to that of work hardening region (3.8 GPa). Thus, the microhardness gap between different areas can also demonstrate the formation of nanocrystallite because it has different deformation mechanism and work hardening behaviors with respect to common coarse grain counterpart [16].

By comparing the microstructure of samples with different coverage, it can be found that the coverage have significant effect on the feature of nano region, although the depth of deformed layer attain a steady value with

Table 1 Processing conditions of air blast shot peening

Processed materials	Material and size of particles	Air pressure (MPa) and speed (m/s)	Coverage (%)
Fe-3.29Si	SUS304 ϕ0.3 mm	0.4, 50–100	3,000, 10,000, 30,000
Fe-0.03C	Fe0.8C ϕ0.8 mm	0.8, 50–100	300, 3,000, 6,000
Fe-0.03C	Fe1.0C ϕ0.05 mm	0.5, 150–200	300, 3,000, 6,000

Fig. 2 Microstructure of as shot peened Fe-3.29Si with different coverage: (**a**) 3,000%, (**b**) 10,000%, (**c**) 30,000%



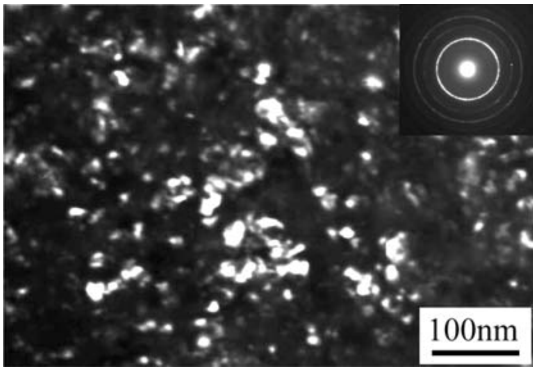


Fig. 3 Nanograins in as treated Fe-3.29Si, the insert is the correspondent SAD pattern

coverage greater than 3,000%. In the sample with lowest coverage 3,000%, only some separated areas like island are formed. In that with coverage 10,000%, more nano regions appeared on the sample surface, while in the sample with highest coverage an almost continuous nanolayer is produced along the sample surface, also its thickness become larger than that in low coverage sample.

The microstructure of samples with subsequent annealing is shown in Fig. 4. It can be seen that the typical recrystallization occurred in the former work hardening region. On the contrary, no obvious change can be detected in nano region by SEM, it indicated that only slight grain growth may be possible to nanograins. The hardness of both regions drop dramatically compared to as-shot state, but the nano region still keeps higher value (3.6 GPa) than that of work hardening (2.9 GPa).

Nanocrystallization in Fe-0.03C

Figure 5 shows the microstructure of shot peened Fe-0.03C by ϕ0.05 mm particles. It can be found that the microstructure near surface consists of two layers clearly. In the order from outer to interior, they are nanocrystallite layer and work hardening layer composed of elongated grains. Although nano regions can be found in all samples with coverage 300%, 3,000% and 6,000%, with the increase of coverage, the amount of nano regions in per unit area rise remarkably, the thickness of work hardening layer also increase slightly.

The microstructure of shot peened Fe-0.03C by ϕ 0.8 mm particles and with subsequent annealing is shown in Fig. 6. Under coverage 300%, the thickness of recrystallization layer, which correspond the former deformed region, is greater than that in the case of ϕ0.8 mm particles. But no nano area is formed yet. Under coverage 3,000% and 6,000%, the thickness of deformed layer in both samples can attain 100 µm roughly, although nano regions can be found occasionally, they only account for very low percentage of the whole deformed layer, which is mainly composed of work hardening region.

Discussions

Nanocrystallization and thermal stability

During the shot peening process, many pits on the surface are formed with hit of particles. The more important thing

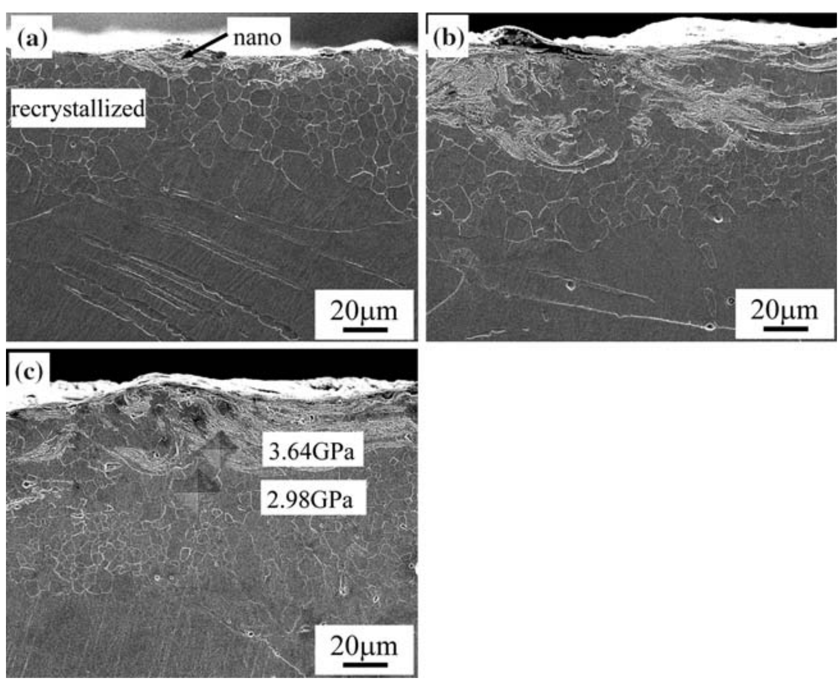


Fig. 4 Microstructure of shot peened Fe-3.29Si after annealing at 873 K for 3.6 ks: (**a**) 3,000%, (**b**) 10,000%, (**c**) 30,000%

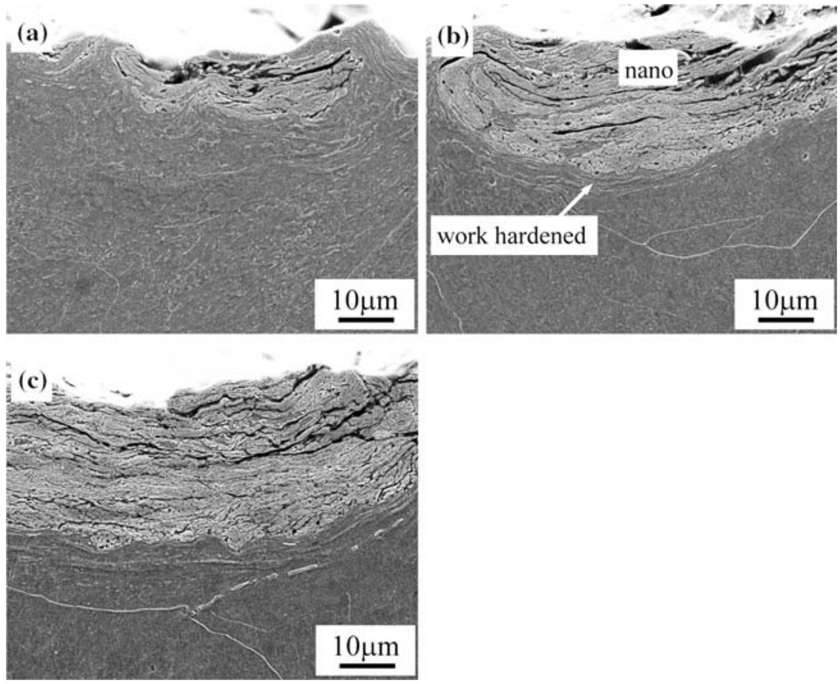


Fig. 5 Microstructure of shot peened Fe-0.03C by ϕ0.05 mm particles: (**a**) 300%, (**b**) 3,000%, (**c**) 6,000%

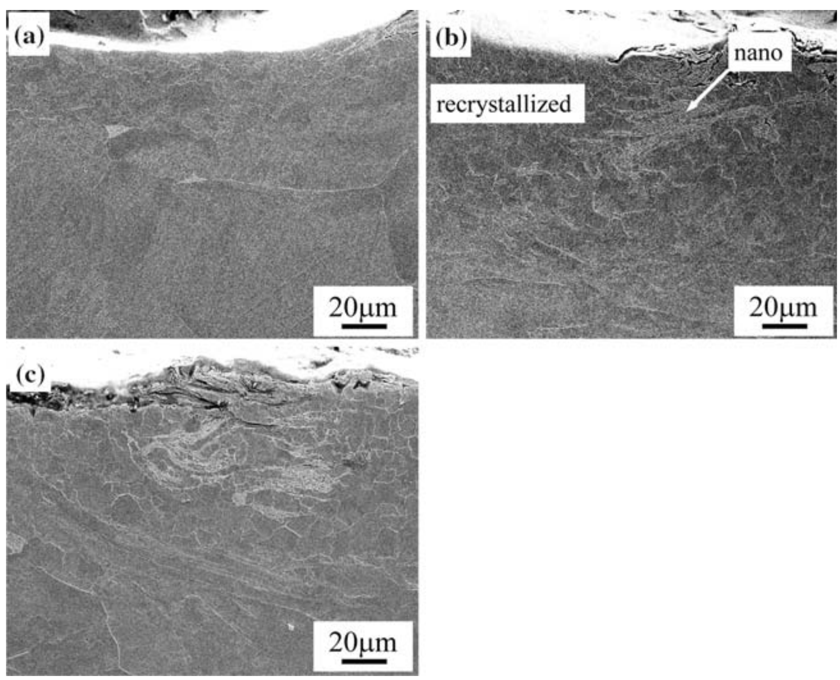


Fig. 6 Microstructure of shot peened Fe-0.03C by ϕ0.8 mm particles with annealing at 873 K for 3.6 ks: (**a**) 300%, (**b**) 3,000%, (**c**) 6,000%

is the formation of extruded ridge around the edge of the pit. When the ridge is hit by another particle, the contact area between sample and particle can decrease significantly, therefore the strain and strain rate can be enhanced dramatically. Additionally, the collision mode also changed from single direction to multiple directions due to the ridge, which is more favorable to the accumulation of dislocations. With the proceeding of collision, some areas will approach the critical condition of nanocrystallization after one suitable hit [17], then some nano regions with clear boundary to work hardening area are formed abruptly, also the depth of deformation layer gradually reach a constant value determined by process condition and material.

Similar to the nanograins obtained by other method [18], the nanostructure produced by shot peening in Fe-3.29Si also shows rather good thermal stability. To nanograins, they are usually dislocation free, which suppress the occurrence of recrystallization. However, the grain coalescence due to grain rotation might be the responsible mechanism to slight grain growth in nanocrystallite.

Effect of processing condition

The nano regions change from discrete area to nearly continuous layer or increase in amount with the increase of coverage. The thickness of whole deformation layer has no obvious change, which depend on the material and the kinetic energy of particles. The size of particle also has important influence on the microstructure. In Fe-0.03C, there is only very fewer nano region is formed in the case of 0.8 mm particles in those two higher coverage despite forming thicker deformed layer than that by 0.05 mm particles. When collide with sample surface, although bigger particles have higher energy, but the contact area also rise at same time, then the strain rate with bigger particles is smaller than that in the case of smaller particles. Consequently, much thicker deformed layer and plain surface is formed by 0.8 mm particles, while nano regions are relative easily produced by 0.05 mm particles.

The main purpose of this processing is to obtain a protective layer on the surface of material, but the surface become rough even some undesirable cracking appear on the top surface after shot peening at certain condition. As shown above, the bigger particles can produce plainer surface, so bigger particles can be used to improve the smoothness of surface after nanocrystallization by small particles. On the other hand, slight abrading can also be performed to remove the cracking while keep the good nanolayer.

Conclusion

Nanocrystallite is successfully synthesized in Fe-3.29Si and Fe-0.03C by air blast shot peening, which demonstrate that it is a simple and useful method to produce nanocrystallite and has potential application. The produced nanocrystallite has extremely high hardness than that of work hardening region, which also has sharp boundary to work hardening area. The nanostructure can keep good thermal stability up to 873 K in contrast to complete recrystallization in work hardening area. The enhanced strain arising from the edge of pit produced by each hit can advance the formation of nanocrystallite. The smaller particles are favorable to nanocrystallization due to higher strain rate resulted from smaller contact area.

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