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Enhanced strength and plasticity of gas nitrided iron by surface mechanical attrition pretreatment

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ABSTRACT

Nitriding treatment at 520 °C for 6 h was performed on a pure iron plate (1.5 mm thick) with nanostructured surface layer, produced by surface mechanical attrition treatment (SMAT). Microstructure, surface hardness and tensile behavior of SMAT nitrided sample were investigated compared to those of coarse-grained nitrided sample. Experimental results showed that a much thicker compound layer and a unique transition zone were developed on the SMAT nitrided sample, leading to an obvious enhancement in surface hardness. Tensile tests revealed that the SMAT nitrided sample exhibits an ultimate tensile strength of ~450 MPa with a total elongation of ~44%, which is much higher than that of the coarse-grained nitrided counterpart (390 MPa, 18%). The higher tensile strength of the SMAT nitrided sample may originate from the contribution of a much thicker compound layer and a unique transition zone. The extraordinary tensile plasticity may benefit from that the precipitation of needle-like nitrides layer was hindered on the SMAT nitrided sample. This enhanced processing method demonstrates the technological significance of nanomaterials in improving traditional processing techniques and provides a new approach to obtain ferrous materials with excellent surface properties and outstanding global tensile properties simultaneously.

1. Introduction

Nitriding is one of the most widely used surface modification techniques to improve surface hardness, wear resistance and anti-corrosion of ferrous materials by the formation of a unique composite structure with a hard compound layer (a layer of ε-$Fe_{2-3}N$ and γ′-Fe_4N compounds) and a diffusion zone (a layer of α-(Fe, N) and needle-like nitrides) [1–3]. However, the nitrided layer formed in the nitriding process is usually accompanied by substantially low toughness [4–8]. The brittleness is believed to be an intrinsic "Achilles' heel" of the nitrided layer. Consequently, the thin ferrous alloy plate usually exhibits a high strength and a limited tensile plasticity after nitriding treatment, which may affect its performance subjected to severe service environments involving high shear, compression and/or impact loading conditions [4,9]. Therefore, the development of a novel nitriding technology to obtain nitrided components with high strength and without sacrificing tensile plasticity, as well as with outstanding surface properties presents a major challenge to engineers and researchers.

A recently developed technique, the surface mechanical attrition treatment (SMAT) used as pretreatment seems to be appropriate for enhancing the nitriding quality efficiently [10–14]. Using SMAT, the nanostructured surface layer can be formed on metallic materials by severe plastic deformation [15,16]. The diffusion behavior in SMAT sample with nanostructured surface layer is considered to be very different from that in coarse-grained sample, owing to the existence of a large number of grain boundaries, which may facilitate atomic diffusion and chemical reactivity [10–14]. For instance, results in our previous study showed that a much thicker compound layer was fabricated on the SMAT iron sample after nitriding at 500 °C when compared with the un-SMAT sample treated under the same conditions [11]. Meanwhile, the structure of overall nitrided layer (including compound layer and diffusion layer) formed on the SMAT nitrided sample is distinct from that on the conventional nitrided sample and can be characterized to three zones form the surface to the inside: the first is a compound layer composed of nano-grained ε-$Fe_{2-3}N$ and γ′-Fe_4N phases, the second is a transition zone with severe plastic deformation, in which the particle-like γ′-Fe_4N phase or other nitrides with submicro grain size was precipitated at grain boundaries (or junctions) of the ultrafine-grained α-Fe phase, dislocations and other defects, and the third is a diffusion zone including α-(Fe, N) phase and small amounts of needle-like nitrides [11]. The surface properties tests illustrated that the surface hardness and wear resistance of the SMAT nitrided sample were greatly improved relative to the coarse-grained nitrided sample [11]. Obviously, this nitrided layer with special structure will bring the positive or negative influence on strength and tensile plasticity of the

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nitrided sample, especially for those of the thin components. Unfortunately, few studies on the tensile properties of SMAT nitrided sample can be found at present. In this work, the tensile properties of the SMAT nitrided iron sample were evaluated in comparison with those of the coarse-grained nitrided sample and the possible mechanisms were discussed.

2. Experiment

2.1. Sample preparation

Elemental iron plate ($1.6 \times 100 \times 100$ mm^3 in size) with a purity of 99.5% was used for this investigation. The as-received sample was annealed at 950 °C for 1 h in vacuum to eliminate the effect of mechanical deformation and to obtain homogeneous coarse grains, followed by the SMAT. The details of set-up and SMAT processing were described previously [15,16]. The basic principle of the SMAT is the generation of a plastic deformation in the top surface layer of treated sample by means of repeated multidirectional impacts of flying balls on the sample surface. The plastic deformation with a large strain and a high strain rate results in a progressive refinement of coarse grains into nanometer regime. In this work, both sides of sample surface were treated for 60 min by GCr15 steel balls (8 mm in diameter) under a vibrating frequency of 50 Hz in vacuum condition, respectively. After SMAT, the thickness of sample was decreased from 1.6 to 1.5 mm due to the severe plastic deformation. Additionally, no contamination was detected on the surface layer by XRD analysis. The cross-sectional microstructures of the SMAT sample are similar with that presented in our previous study [11]. In brief, a nanostructured surface layer of about 20 μm thick was fabricated on the top surface layer. The average grain size in the top surface layer is about 8 nm. Meanwhile, a severe plastic deformation layer, nearly 40 μm thick was detected between the nanocrystalline layer and matrix, in which the average grain size increases gradually with depth from the treated surface.

The SMAT samples were cut into small dimensions ($1.5 \times 30 \times 30$ mm^3 in size) and were ultrasonically cleaned in ethanol. Then the SMAT samples were nitrided in a homemade device with flowing NH_3 at 520 °C for 6 h under 1.5 atm pressure. For comparison, the coarse-grained samples were also nitrided under the same conditions. After nitriding for required time, the samples were cooled to room temperature by slow furnace cooling.

2.2. Structure characterization

Cross-sectional structural characterizations of the processed samples were carried out on a Leica DMR optical microscope (OM) and a JSM-6301F scanning electron microscope (SEM). Nitrogen distribution in the nitrided surface layer was monitored by an Oxford INCA X-ray energy dispersive spectroscope (EDS). Microstructure characterizations were also examined by using a Jeol-4000FX transmission electron microscope (TEM) with an operating voltage of 200 kV. The samples for TEM observation were prepared by grinding and mechanical polishing followed by ion-thinning at lower temperature. The phase in the surface layer was identified by means of PW3040/60 X'Pert Pro X-ray diffraction (XRD) using Cu Kα radiation (40 kV, 200 mA).

2.3. Hardness and tensile properties

The microhardness variations along depth in the samples were measured by using a L101MVD model Vickers microhardness tester with a load of 15 g and a testing time of 10 s [17]. Uniaxial tensile tests were performed on an Instron 5848 machine at a strain rate of 6×10^{-3}/s. Non-standard dog-bone shaped tensile samples with a gage cross-section of 6×1.5 mm^2 and a gage length of 10 mm were used. The schematic drawing and dimensions were shown in Fig. 1. In this paper, the purpose of an unusual tensile specimen design is to obtain a cross-section. Subsequently, this cross-section was chemically polished to observe deformation trace which may formed during tensile testing at different strain level. The observation of deformation trace may help us to compare the tensile plasticity directly between the SMAT nitrided sample and coarse-grained nitrided sample. The displacement of the tensile gage section was measured using a laser extensometer with an accuracy of 1 μm. The fracture surface observations were conducted by the JSM-6301F scanning electron microscope.

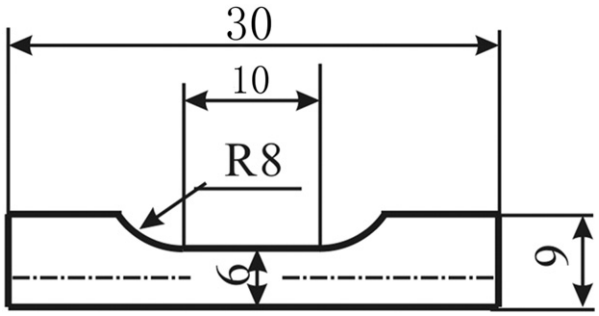

Fig. 1. Schematic drawing and dimension of the tensile specimen (mm).

3. Results

3.1. Microstructure characterization

After nitriding, the microstructure of nitrided layer formed on the SMAT nitrided and coarse-grained samples was characterized systematically. For the SMAT nitrided sample, the nitrided layer includes compound layer, transition zone and diffusion layer from the surface to the inside, respectively, as shown in Fig. 2(b). However, The nitrided layer on the coarse-grained sample is generally subdivided into a compound layer and a diffusion zone, as shown in Fig. 2(a). Meanwhile, the compound layer thickness in the SMAT nitrided sample is ~30 μm, evidently larger than that in the coarse-grained sample (~17 μm). Additionally, the severe plastic deformation layer (transition zone) was retained underneath the compound layer on the SMAT nitrided sample, suggesting that the nanostructured surface layer formed by SMAT exhibits good thermal stability during nitriding treatment. Specially, the needle-like nitrides (γ′-Fe_4N) precipitated in diffusion layer of the SMAT nitrided sample is shorter than that in the coarse-grained nitrided sample and grew along the plastic deformation trace. Further TEM observations combined electron diffraction analyses showed that the compound layer of the SMAT nitrided sample is composed of ultrafine polycrystalline ε-$Fe_{2-3}N$ and γ′-Fe_4N phase nitrides (see Fig. 2(c)). Additionally, a large amount particle-like γ′-Fe_4N phases with submicron grain size were precipitated underneath the compound layer, as shown in Fig. 2(d). With increasing depth, the grain size of particle-like nitrides in the transition zone increases gradually, as shown in Fig. 2(e). In summary, the structure of entire nitrided zone is similar with that on the SMAT sample nitrided at 500 °C for 2 h [11]. The only difference is that the thickness of nitrided layer was increased when nitriding at higher temperature and for a longer duration. The mechanisms of strongly enhanced diffusion kinetics in iron by means of SMAT have already been demonstrated in literature [10–12,14,18–20]. Concisely, a large volume fraction of grain boundaries with a high excess stored energy promotes the diffusion of nitrogen atoms and increases the nucleation and growth rates of compounds in the nanostructured surface layer induced by SMAT.

3.2. Hardness

The hardness distributions along the depth of SMAT nitrided sample and coarse-grained sample are shown in Fig. 3. Due to the formation of thicker compound layer with nano-grained nitrides and the fabrication