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## Dynamic recrystallization of Fe-Cr alloys by atmospheric-controlled induction-heating fine particle peening



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#### ABSTRACT

Atmospheric-controlled induction-heating fine particle peening (AIH-FPP) was conducted in a N<sub>2</sub> atmosphere to form ultrafine grains on the surface of Fe-Cr alloys. The surface microstructure of Fe-2%Cr alloy and Fe-10%Cr alloy treated with AIH-FPP at 973 K and 1073 K was characterized using optical microscopy, scanning electron microscopy (SEM), electron backscatter diffraction (EBSD), and transmission electron microscopy (TEM). AIH-FPP can be used as a thermomechanical process to form ultrafine grains on the surface of Fe-Cr alloys because strain is induced by AIH-FPP at a high rate and high temperature during dynamic recrystallization. In particular, the grain size of Fe-Cr alloys tends to decrease with increasing AIH-FPP treatment time. This is attributed to the increase in the strain induced by AIH-FPP with increasing AIH-FPP treatment time during dynamic recrystallization. Thus, the AIH-FPP-induced stratification structure composed of fine grains becomes more pronounced with increasing treatment time. Furthermore, the AIH-FPP treatment temperature at which fine grains are formed depends on the Cr concentration of the Fe-Cr alloys.

#### 1. Introduction

Metals are used under severe conditions in a variety of engineering applications, necessitating the improvement of their various properties, such as mechanical strength, tribological properties, and corrosion resistance. The addition of alloying elements to metals is particularly effective in improving their properties. For example, a new type of austenitic stainless steel, super-austenitic stainless steel, which has higher elemental concentrations than conventional steels and a superior corrosion resistance, has been developed by adding chromium, molybdenum, and nickel to conventional steels [1]. However, stainless steels are costlier than structural steels, owing to the large amount of added rare metals.

Therefore, grain refinement processes have been introduced to improve the mechanical properties of ferrous materials, including Fe-Cr alloys, which are the focus of the present study. Meng et al. [2] reported that passive films of Fe-10%Cr alloys could be enriched with Cr more easily as a result of grain refinement, which provides diffusion paths for Cr extending to the surface and the resulting films were also passivated more easily. Gupta et al. [3] reported that nanocrystalline Fe-10%Cr alloys, fabricated by high-energy ball milling, followed by compaction and sintering, exhibited superior oxidation resistance, owing to the formation of a more protective oxide scale. In particular, two types of treatments are effective in forming fine grains of steel: (i) severe plastic deformation (SPD) processes such as ball milling [3], high-pressure torsion (HPT) [4,5], high-speed drilling [6], surface mechanical attrition treatment (SMAT) [7-9], and shot peening [10-13], and (ii) thermomechanical processing using recrystallization-controlled rolling [14], multipass hot deformation [15], and warm peening [16]. For the purposes of thermomechanical processing, metallic materials are categorized into dynamic recrystallization-type materials, such as nickel, copper, and austenitic stainless steel with fcc structure, or dynamic recovery-type materials, such as ferritic iron with bcc structure, which is the focus of the present study [17]. Dynamic recrystallization was observed in ferritic iron [17-20]; however, Tsuji et al. [17] found that some grains in ferritic iron hardly recrystallized even in small-Zener-Hollomon-parameter (Z) deformations [21], owing to the initial orientation dependence of recrystallization in the ferrite. Therefore, surface modification processes that can be performed through large-Z deformations in ferrous materials must be designed.

Against this background, we developed a surface treatment system that combined a high-frequency induction heating (IH) system with fine

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Received 22 December 2017; Received in revised form 10 March 2018; Accepted 13 March 2018 Available online 13 March 2018 0257-8972/ © 2018 Published by Elsevier B.V. particle peening (FPP), hereafter abbreviated as IH-FPP [22,23]. IH-FPP can deform a material's surface at high temperature in austenite region, which results in the formation of fine grains of martensitic stainless steel [22] and chromium-molybdenum steel [23]. This is because the particle velocity obtained by FPP is higher than that obtained by conventional shot peening [24,25], and thus, a large number of particles impact the surface within a short period during IH-FPP. Furthermore, the present authors have previously modified the IH-FPP system through the suppression of oxidation by controlling the processing atmosphere [26,27]. Atmospheric-controlled IH-FPP (hereafter "AIH-FPP") can effectively improve material properties by modifying the microstructure of the material surface and may be employed as thermomechanical processing in the case of steels.

The purpose of the present study was to form ultrafine grains of Fe-Cr alloys with bcc structure by AIH-FPP in a  $N_2$  atmosphere. In particular, AIH-FPP was performed for Fe-Cr alloys with no C element to clearly examine the effects of Cr element on the microstructure of ferrous materials by the AIH-FPP in the present study. Furthermore, the surface microstructure of Fe-Cr alloys treated with AIH-FPP is characterized, and the mechanism for the microstructural change of AIH-FPP-treated Fe-Cr alloys is discussed.

#### 2. Experimental

#### 2.1. Materials and specimens

This study employed two types of Fe-Cr alloys containing 1.98% Cr and 10.00% Cr (mass%), with the balance being Fe (hereafter Fe-2%Cr and Fe-10%Cr, respectively), fabricated by arc melting. These materials were hot-rolled at 1273 K under a rolling reduction of 50%, and then heated at 1273 K for 1.8 ks and cooled in the furnace. The average grain size, which was calculated by the intercept method based on the optical micrographs of Fe-2%Cr and Fe-10%Cr, were 241 µm and 137 µm, respectively. Initial grain sizes of Fe-Cr alloys are different because the continuous cooling transformation (CCT) diagrams depend on the Cr concentration. The Vickers hardness of Fe-2%Cr and Fe-10%Cr were 105.2  $\pm$  2.9 HV and 127.4  $\pm$  4.4 HV, respectively (*n* = 20). Materials with dimensions of 9 × 80 × 150 mm were machined into cylindrical specimens with 15 mm diameter. After machining, the specimens were polished with emery paper to a thickness of 4 mm and a mirror finish.

#### 2.2. AIH-FPP treatment

AIH-FPP was performed on the mirror-finished specimens in a N2 atmosphere to form fine grains by using the developed treatment system, the details of which can be found elsewhere [26,27]. Fig. 1 illustrates the thermal history during AIH-FPP. AIH-FPP can induce strain at the material surface at high temperature without oxidation. The polished specimen was placed in the IH coil, and the atmosphere in the chamber was purged by supplying N2 gas through the peening nozzle. When the oxygen meter (measurement tolerance: 0.3 vol%) in the chamber showed 0.0 vol%, AIH-FPP was performed at a peening pressure of 0.5 MPa, a particle supply rate of 1.0 g/s, and a nozzle distance of 100 mm. Shot particles with 150-180 µm diameters were used and were prepared by a high-speed tool steel with a chemical composition (mass%) of 1.3% C, 4.0% Cr, 5.0% Mo, 3.0% V, 6.0% W, and 8.0% Co, with the balance being Fe. Fig. 2 shows an example of thermal history for AIH-FPP at 973 K for 120 s. The temperature was measured on the top surface of specimens by using a thermocouple during AIH-FPP, which was conducted at 973 K and 1073 K for 30, 60, 120, and 180 s. The specimens were rapidly cooled by compressed air after supplying the shot particles.

#### 2.3. Microstructural characterization

The surface microstructure of the AIH-FPP-treated specimens was



Fig. 1. Schematic of thermal history during AIH-FPP.



Fig. 2. Thermal history during AIH-FPP (treatment temperature: 973 K, treatment time: 120 s).

characterized using optical microscopy, scanning electron microscopy (SEM), electron backscatter diffraction (EBSD) in an area of  $44.0 \times 58.6 \ \mu\text{m}^2$  observed at  $2000 \times$  magnification under the condition of  $0.23 \ \mu\text{m}$  in a step size at an accelerating voltage of  $15 \ \text{kV}$  in  $15 \ \text{mm}$  of working distance, and transmission electron microscopy (TEM) at an accelerating voltage of  $200 \ \text{kV}$ . In the present study, the grain size was calculated by the intercept method based on the optical micrographs of Fe-2%Cr and Fe-10%Cr after etching with Nital or Vilella solution. The hardness distributions were measured along the longitudinal sections of the AIH-FPP-treated specimens using a micro Vickers hardness tester at a load of  $0.245 \ \text{N}$  and a holding time of  $25 \ \text{s}$ . The distances between indents were  $100 \ \mu\text{m}$  in the present study to eliminate the effect of plastic deformation in the hardness tests on the hardness value in the subsequent hardness tests.

#### 3. Results and discussion

## 3.1. Effect of treatment temperature during AIH-FPP on microstructure of Fe-Cr alloys

To examine the effect of treatment temperature during AIH-FPP on the microstructure of Fe-Cr alloys, the surface microstructures of Fe-Cr alloys treated with AIH-FPP at various temperatures for 60 s were Download English Version:

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