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Strain-Induced Diffusion of Nickel in Nanocrystalline Fe Produced by High Energy Shot Peening

Sun Jianchun, Chen Dengming, Ma Yilong

Chongqing University of Science and Technology, Chongqing 401331, China

Abstract: A nanocrystalline surface layer without oxidation, porosity or contamination was obtained by high energy shot peening (HESP) to a pure iron cylinder. The thickness of the nanocrystalline surface layer was more than 100 μ m and the average grain size was about 50 nm. The pulse pressure diffusion (PPD) and constant pressure diffusion (CPD) were used to diffuse nickel into nanocrystalline iron at 850 °C on Gleeble 1500. Results show that the diffusion coefficient of Ni in the nanocrystalline Fe is 1 order of magnitude higher than that in coarse-grained Fe and 1 order of magnitude higher than that using CPD. The enhanced diffusivity of Ni may originate from the following three reasons: (1) pulse pressure can break the metallic compound formed during diffusing which may block the diffusion of Ni to inner nanocrystalline Fe; (2) a considerable amount of triple junctions and (3) a large volume fraction of non-equilibrium grain boundaries (GBs) in the present nanocrystalline Fe sample processed by the HESP technique.

Key words: nanocrystalline Fe; high energy shot peening; diffusion coefficient; nickel atom

During the fabrication and treatment of materials, such as nitridation, surface alloying and diffusion bounding, an enhanced diffusivity of atoms at low temperature and pressure is always wanted. According to the diffusion theory, to enhance the diffusion coefficient at a fixed or lower temperature, there must be more diffusion channels in solids. The difference between nanocrystalline materials and the conventional polycrystalline systems is the increased volume fraction of grain boundary region which makes the nanocrystalline materials to exhibit, in general, an enhanced atomic diffusion. It has been reported by the experimental results in the literature^[1-3]. However nanomaterials prepared by current methods, such as gas phase condensation, mechanical alloving and sputtering, always contain impurities and holes, which may hinder the diffusion of atoms in them. So bulk nanocrystalline solids without porosity and contamination are always expected to be prepared for the study of the diffusion of atoms in nanocrystalline materials.

Because the atomic diffusion in nanocrystalline is mainly the grain-boundary diffusion^[4-7], one main direct way to

enhance the diffusivity of atoms in them is refining the grains to increase the fraction of grain boundaries, which can act as diffusion channels. But to a fixed nanomaterial, loading a pressure on the diffusion couple can help to improve the movement speed of the atoms of diffusion source and an atomic level contact of the diffusion couples. Here two different pressures, constant pressure and pulse pressure, were loaded on the iron and nickel diffusion couple, and the diffusion processes were called constant pressure diffusion (CPD) and pulse pressure diffusion (PPD) respectively. Due to a high activation of atoms in nanocrystalline materials, when the atoms exchanged from two different materials into each other, metallic compounds always formed on the interface, which on some extend would prevent the further diffusion of atoms^[8-10]. A pulse pressure exerted on the xenogenic metallic materials has been proved to be an effective way to delete the metallic compound formed during the diffusion^[11]. In the present work, the iron-nickel diffusion couples were subjected to a pulse pressure to enhance the diffusivity of nickel atoms in iron, and we defined the diffusion as strain-induced diffusion.

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Corresponding author: Sun Jianchun, Ph. D., Associate Professor, College of Metallurgy and Materials Engineering, Chongqing University of Science and Technology, Chongqing 401331, P. R. China, Tel: 0086-23-65023714, E-mail: kwensun@163.com

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1 Experiment

An iron cylinder ($\Phi 10 \text{ mm}$ and $\Phi 30 \text{ mm}$) with a purity of 99.95% and homogeneous coarse grains of α -Fe phase (100 µm, in Fig.1) was subjected to the high energy shot peening (HESP) processing to get a nanocrystalline surface layer. Before HESP treatment the Fe sample was polished and fully annealed at 800 °C in a vacuum heat treatment furnace for 200 min to eliminate the effect of inner stress and mechanical polishing on the surface structure.

During the HESP procedure, hardened cast iron balls (with a mirror-like surface and a diameter of 0.5 mm) were placed at the bottom of shot tank (Fig.2). When the compressed gas was blown into the working tanker from the peening gun, the balls were accelerated and got a high kinetic energy. After the balls with a high kinetic energy reached the surface of the sample, the kinetic energy would be transformed into plastic deformation in the samples. The balls after the release of energy were absorbed into the gun again because of a negative pressure and this made the HESP procedure recycling, which would lead to an accumulation of deformation in the samples. The accumulated deformation in the sample, and it may cause grain refinement of the sample.

Microscopy and SEM were used to examine the microstructure features of the nanocrystalline layers. Meanwhile EDS was used to determine the diffusion depth and Ni contamination in nanocrystalline Fe. The X-ray diffraction (XRD) was used to study the structure evolution of the layer of the Fe sample with Cu K α radiation. Small angular steps 2 θ =0.03° were taken to measure the intensity of each Bragg diffraction peak. The counting time of 20 s was used to measure the width of diffraction peak in the step-scanning mode. The average grain size and the mean microstrain of the nanocrystalline Fe layer were derived from the line broadening of BCC Fe (110), (200) and (211) Bragg diffraction peaks by Scherrer method.

The diffusion of nickel in nanocrystalline and coarse Fe was carried on the Gleeble-1500D thermal simulation machine which can provide a controllable temperature and pressure in a vacuum environment. To compare the diffusion of nickel in both nanocrystalline and coarse Fe, we designed an assembly of diffusion samples, as shown in Fig.3, which can provide the



Fig.2 Schematic illustration of the HESP treatment set-up

same diffusion parameters for nickel in both nanocrystalline and coarse Fe.

Pure nickel foil with a thickness of 13 μ m was used as the diffusion source, see in Fig.3. The diffusion was conducted at 850 °C within 20 min under a constant pressure (CP, F=10 MPa) and a pulse pressure (PP, $F_{max}=16$, $F_{min}=8$, f=0.5 Hz).

2 Results and Discussion

2.1 Microstructure characterization of the HESPed Fe

Detailed microstructure of the surface layer was characterized by SEM observation of the cross-section of the peened samples. Fig.4 shows the full view of HESPed Fe after 7 min HESP treatment and the high magnification of the top surface layer. As shown in Fig.4, the whole deformed layer with a thickness about 120 µm contain three different parts: the severely deformed part on the top surface in which the grains are extremely refined (marked as A), the deformed layer in which the grains are refined to some extent (marked as B) and the transitional region in which the grains are yet not refined and just a little deformation occurs (marked as C). But there are no obvious interfaces between the 3 regions, so here we call the whole deformed layer as HESPed layer. As shown in the high SEM magnification of the top surface layer, the grains cannot be separated from each other because there are no obvious boundaries of the iron, which indicates HESP treatment can refine the iron grains. Besides the grain orientation has been changed and fiber structure forms because of HESP treatment, but whether the grains are nanosized or not is uncertain. The



Fig.1 Metallurgical structure of iron after annealing



Coarse iron with a nanocrystalline layer

Fig.3 Assembly of diffusion samples

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