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Nitridation of iron by CW-CO_2 laser nitriding technologies

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Abstract

Laser nitriding is an attractive method to improve metal surface properties, such as hardness, wear, and corrosion resistance, with the advantage of simple experimental setup, rapid treatment, and precise process control. Here, iron nitride (Fe_xN) is obtained by the mixing technology with laser and pre-heated nitrogen (LHN) or nitrogen plasma (LPN) beams on the surface of pure iron in atmosphere ambient. In these two technologies, laser provides heat, pre-heated nitrogen provides help for nitrogen activity, and nitrogen plasma provides nitrogen ions, respectively. The two technologies are superior to the previous laser nitriding (LN) methods. However, iron nitride more difficult was obtained by the mixing technology with laser and pre-heated nitrogen than by the mixing technology with laser and plasma, and iron oxide was not observed in the sample treated by the latter. The feasibility of the technologies is analyzed in theory. X-ray diffraction measurements reveal formation of iron nitride in the as-treated sample by these technologies. Moreover, the concentration and phase formation of iron nitride were given at different laser power densities and different scanning velocities with emphasis on the mixing technology with laser and plasma.

Keywords: Iron nitrides; Laser and nitrogen; Laser and plasma; Surface modification; Laser nitriding

1. Introduction

Nitridation of iron has been a subject of great importance in the context of diversified applications in mechanical industry, because nitridation is known to modify such properties as wear resistance, friction, corrosion, fracture toughness, micro-hardness, and so on, significantly [1–5]. There are many methods for nitriding, for example salt bath nitriding [6], reactive magnetron sputtering [7], plasma nitriding [8], plasma immersion ion implantation [9] and laser nitriding [3,10–13]. In recent years, laser-induced chemical reactions in particular are becoming more and more popular. It is well established that the irradiation of iron and other metals with excimer or CO_2 laser in a nitrogen atmosphere or air [3,14,15] leads to a huge take-up of nitrogen into the irradiated surfaces. The laser nitriding effect has been demonstrated for various materials and for different laser systems. For laser nitride, the smaller heat-affected zone and mass-less nature of laser light present a unique way of nitriding surface of substrates that are sensitive to elevated temperatures or to particle bombardment. However, the laser nitriding technology requires very high energy density, so the cost is high. Recently, there has been a considerable emphasis on exploring ways to achieve low-energy nitridation of the surface layers of industrial metals and alloys in atmosphere [16]. Only few studies have been reported so far on the mixing technology of laser and pre-heated nitrogen (LHN) or plasma nitriding (LPN) of Fe_xN. With pre-heated nitrogen providing more active nitrogen or nitrogen plasma beam providing the ions source for the reaction, the two technologies differ significantly from the conventional method and the active nitrogen or ions of low-flux nitrogen plasma lead to a stronger take-up of nitrogen into the iron and nitride formation. And the mixing technology with laser and nitrogen plasma is superior to the mixing technology with laser and pre-heated nitrogen. Towards this end, the technique of laser and nitrogen plasma implantation will be very useful and easier to obtain iron nitride. Since the laser and plasma beams coaxially process the surface layers of metals simultaneously, laser power density can be effectively decreased to 10^5 W/cm^2 and oxidation can be rejected in atmosphere by LPN. Therefore,

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it is important alternative way to nitride material surfaces, which could be novel, simple, efficient, and compatible technology.

In this paper, we report on nitridation of iron by the different laser nitriding technologies and compare the nitriding effect of LPN with the traditional technology of laser nitriding (LN) and LHN in atmosphere. The micro-structural characterization of the state of nitride thus was formed by new technologies. It is important to prove the feasibility of above new method in applications. This work is based on recent studies of laser-induced reactive quenching at the liquid–solid interface, a new process for synthesis of metastable compound films [17].

2. Experiment details

The processing laser is a 500 W CW-CO_2 laser, and power density up to 5×10^6 W/cm^2 could be obtained within an acceptable degree of homogeneity by suitable beam guiding and focusing to a spot diameter size of 0.3 mm. Shown in Fig. 1 are the schematic diagrams of the experimental system. The laser can be reflected onto the surface of sample by the speculum. In the treatment by LHN, the nitrogen was heated by an electric stove and sent to the surface of sample coaxially together with laser beam. However, in the treatment by LPN, the plasma gun was used. Plasma gun was fixed on the scanning worktable. An alternating current launcher, in our case, generates, at 50 Hz, $a \leq 10$ kV that creates and sustains the discharge between two parallel electrodes. The inside diameter of the hollow electrodes is 4 mm. The focused laser can pass through it and heat the surface of sample. The plasma torch stems from the species generated by the plasma and carried out, by the gas flow, out from the hole in the cathode. These species consist mainly of ionic, atomic, molecular nitrogen and electron. The current intensity to the discharge is limited using a low concentration impedance matching network. The maximum current delivered to the discharge is limited to 4 mA. The flow of N_2 is regulated using LZB flow-meters, which allow for a maximum flow of 1.0 m^3/h.

The scanning worktable can let the laser and pre-heated N_2 or plasma spots scan over the sample surface to obtain an effective modified area of 20 mm × 14 mm. The samples are 0.3 mm thickness pure iron (Fe > 99.85 wt.%). Their surfaces were abraded and cleaned ultrasonically in three solvents subsequently: trichloroethane, acetone, and methanol.

The X-ray diffraction patterns for the virgin and treated foils in different conditions were recorded on a machine. The X-ray source is Cu Kα under 40 kV and 50 mA, the recording step is 0.02° and the scanning speed is 8°/min. The elliptically polarized light method is used to obtain the thickness of the nitride film.

3. Results and discussion

3.1. Comparison with different technologies

In the experiments, laser power density ($I = 1 \times 10^6$ W/cm^2) and nitrogen flux ($F = 1.0$ m^3/h) was fixed and the sample was treated by different CW-CO_2 laser nitriding technologies at the value of 0.1 mm of scanning space and the value of 100 mm/min of scanning velocity.

In order to compare the effect on the iron with different technologies, we performed the small-angle X-ray diffraction (XRD) measurements. The results are shown in Fig. 2 with a representing the untreated sample, b the sample treated by LN, c the sample treated by LHN and d the sample treated by LPN. The pattern of Fig. 2a shows the α-Fe contribution. The line intensity ratio exhibits only the peaks of the original

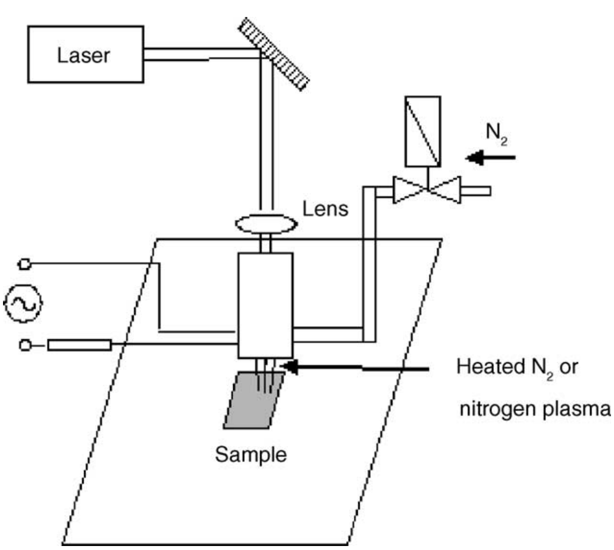

Fig. 1. Schematic of the plasma gun.

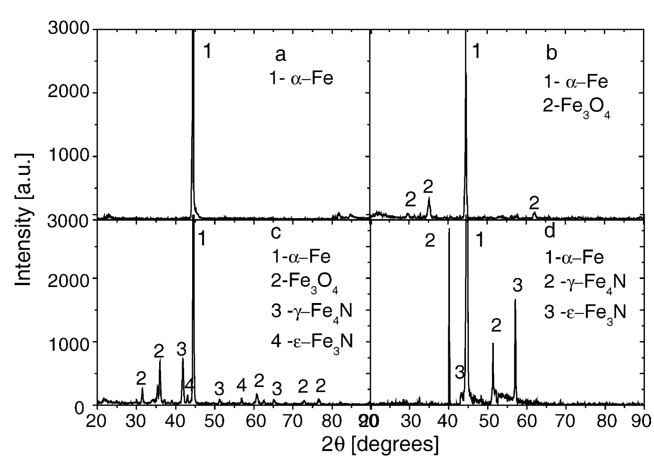

Fig. 2. X-ray diffraction patterns for: (a) the untreated sample, (b) the sample treated by only laser, (c) the sample treated by laser and pre-heated nitrogen, and (d) the sample treated by laser and nitrogen plasma.