Note

Fabrication of Micropore on AISI 304L Austenitic Stainless Steel Surface by High-current Pulsed Electron Beams Irradiation

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1. Introduction

During the past decade, high-current pulsed electron beams (HCPEB), initially developed at the Tomsk Institute of High Current Electronics,^{1,2)} have attracted much attention in the field of materials surface modifications. Under the action of HCPEB a high energy (10^8 – 10^9 W/cm²) is deposited only in a very thin layer (less than tens of micrometers) within a short time (a few microseconds) and causes superfast heating and cooling on the irradiated surface of material. Unfortunately, craters are inevitably formed on the irradiated surface when metals and alloys are irradiated by HCPEB, which are believed to deteriorate the mechanical reliability in structural materials and limit the applications of this new technique.^{1–4)} However, this technique may become a new method for porous materials preparation by controlling HCPEB process parameter and choosing appropriate materials.

Porous materials have been industrialized into permeable application such as gas filter, separation membranes, and catalyst supports.^{5–8)} For such applications, a high porosity with a well-controlled pore size distribution is required. On the other hand, porous materials with a tailored microstructure have potentials to be used as structural components due to unique properties such as good strain and damage tolerance, and good thermal shock resistance. In practice, some performance based on size, shape, and polarity is difficult

to achieve using polymeric porous materials. Additionally, the thermal and chemical stability of organic materials is often limited. In contrast, metal porous materials can provide unprecedented selectivity and stability.

In this note, we provide an initial assessment of the feasibility of good dispersions of micropores on the surface of AISI 304 austenitic stainless steel by HCPEB treatment. The initial experiments were performed on a small laboratory scale.

2. Experimental

Our experiments were conducted on a HCPEB equipment of Nadezhda-2 type. It produced an electron beam of low electron energy 10–40 kV, high peak current 10^2 – 10^3 A/cm², short pulsed duration 0.5–5 μ s and irradiated area 30 cm². The electron beam is generated by an explosive emission cathode. The accelerating voltage, magnetic fields strength and the anode-collector distance control the beam energy density. For more details about the HCPEB system, see Refs. 1), 2).

AISI 304L austenitic stainless steel with composition of (wt%): 18.2 Cr, 9.4 Ni, 1.5 Mn, 0.4 Si, 0.2 Mo, 0.2 Cu, 0.1 Co, 0.003 Ti, 0.027 P, 0.003 S, and 0.044 C (balance Fe) was selected as the target material. Specimens were machined with a size of 14 mm in length, 10 mm in width, and 6 mm in height, and one side surface was mirror polished. The polished surfaces of samples were irradiated at room temperature with 1 and 10 pulses using this HCPEB source, respectively. The HCPEB irradiation was carried out under the following conditions: the electron energy 28 keV, the current pulse duration 3.5 μ s, the energy density about 4 J cm⁻², and the vacuum 10^{-5} T.

Microstructural examinations were performed with a optical microscope and a transmission electron microscope (TEM) of type H-800. The thin foils for the TEM observation were obtained by mechanical pre-thinning, dimpling, and electrolytical thinning from the substrate side. The TEM microstructures observed were at about 5–15 mm depths from the irradiated surface.

3. Results and Discussions

Our previous works indicated that abundant defect structures including stacking faults, structure of dislocation tangles, twin lamellae and, especially, various vacancy defect clusters were introduced in the metal materials during the course of HCPEB irradiation.^{9–12)} Figure 1 shows the dislocation loops of the reference samples induced by both irra-

Fig. 1. TEM images showing the dislocation loops of the AISI 304L stainless steel samples irradiated at 1 and 10 HCPEB pulses, (a) 1 pulse (b) the dislocation loops close to the twins at 10 pulses at 10 HCPEB pulses.

Fig. 2. TEM images showing the vacancy defect clusters of the AISI 304L stainless steel samples irradiated at 10 HCPEB pulses. (a) TEM dark-field image showing dense small defect clusters, (b) and (c) showing large-size SFT located at the tip of twin lamellae, (c) and (d) showing large-size SFT located inside or in the twin lamellae, (d) voids.

diated conditions. After 1 pulse, large numbers of small dislocation loops (about 20 nm) have been well established as shown in Fig. 1(a). In the case of 10 pulses, more numbers of small dislocation loops were observed. It is worth to note that the dislocation loops located near twin lamellae possess larger sizes which can be estimated directly, vary from 50 to 80 nm approximatively, as shown in Fig. 1(b). Those larger dislocation loops were basically located at the tip and/or the boundary of twin lamellae. During a certain period of time after the start of electron irradiation in TEM, the dislocation loops appear to become little shrink. Based on Kiritani's analysis,¹³⁾ the majority of these dislocation loops observed after HCPEB irradiation were identified as of vacancy type.

Besides the vacancy dislocation loops, both stacking fault tetrahedra (SFTs) and voids were also observed in the sample with 10 pulses irradiation. **Figure 2(a)** shows that a mass of small vacancy clusters were formed after 10 pulses HCPEB irradiation. Especially, the sizes of the SFTs, vary from 100 to 250 nm approximatively and located at the tip of twin lamellae, (Fig. 2(b)), or at the boundary of twin lamellae (Fig. 2(c)) are very large. The relation between SFTs and twin lamella has been reported elsewhere.⁹⁾ Another type of vacancy cluster, voids, was also presented in the regions with high dislocation density as shown in Fig. 2(d).

The formation of a mass of vacancy defect clusters suggests that supersaturation vacancies can be introduced after HCPEB irradiation. It corresponds to Prgrebnjak *et al.*'s result detected by a positron annihilation method. They demonstrated that a high density of non-equilibrium vacancies (up to 10^{-3}) was formed in the near-surface of pure iron irradiated by HCPEB.¹⁴⁾ The density of non-equilibrium vacancies is much higher than that of the situation at room temperature.

In general, the lattice sites in perfect-crystal are occupied completely by natural atoms. Thus, the migration of atoms is very difficult. However, after HCPEB irradiation, the

presence of supersaturation vacancies in irradiated samples provide a possibility for atom diffusion which result in the accumulation of vacancies and the formation of point defect clusters as shown in Fig. 1 and Fig. 2. For the irradiated samples with the considerable defect structures and significant volume fraction of dislocations, grain boundaries and twin boundaries, the excess vacancies can find sinks easily and quickly. Therefore, these vacancies (clusters) are likely to migrate towards the surface because the temperature of the surface layers is increasing with higher rates than the rates of its decrease in an interior part of the sample under the influence of temperature gradients.¹⁴⁾ In this case those one- and two-dimensional defects are likely to serve as preferential paths of vacancy (cluster) migration. As long as vacancies (clusters) migrate to the irradiated surface, vacancy accumulation can occur. As a result, the local density of material decrease near such defects and the formation of the micropores on the irradiated surface can be expected.

Figure 3 shows the typical morphologies of the surface of the irradiated samples treated with 1 and 10 pulses, respectively. The microstructure on the irradiated surface can be observed directly and the chemical etching process is not needed. It can be seen clearly that the irradiated regions of the sample surface become rougher and many craters are formed on it. The craters shown in Figs. 3(a) and 3(b) range from a few μm to 50 μm approximatively in diameter and appear to be distributed uniformly on the surface. According to our previous studies, such a typical morphology is the result of the local sublayer melting and eruption through the solid outer surface.³⁾

Figure 4 represents the magnified surface morphologies of the regions where the craters are seldom presented after 4 J/cm² HCPEB treatment with 10 pulses. The observation of micropores are remarkable over the irradiated surface, as shown in Fig. 4. Figure 4(a) reveals that the micropores with larger sizes (about 1–2 μm) are the preferred form at the regions of original grain boundaries,¹⁵⁾ which corresponds to our analysis mentioned above. In the regions with

Fig. 3. Showing the morphologies of the surface craters after 4 J/cm² HCPEB treatment, (a) 1 pulse, (b) 10 pulses.

Fig. 4. Showing the morphologies of the surface micropores after 10 pulses HCPEB treatment, (a) at the region of original grain boundaries, (b) in the regions with intense deformation.

intense deformation, very high density of micropores with a good dispersion and uniform size distribution and rounded shape are presented on the irradiated surface. Most of the micropores are isolated from each other and the sizes of micropores are directly evaluated, 100–300 nm approximately, from Fig. 4(b). It may be attributed to that HCPEB irradiation causes intense deformation at the surface of material^{10,11)} and leads to the formation of considerable defect structures within the interior of grains, which provide preferential paths of vacancy migration.

4. Conclusions

Our experimental results show the possibility that surface porous metal can be successfully fabricated by using HCPEB technique. The SEM analysis indicate that fine size and good dispersions of micropores can be achieved on the surface of treated material by adjusting HCPEB process parameter and selecting appropriate materials. If we choose the sample with appropriate thickness, the thicker porous coating of metal or alloy (or inorganic materials) may be successfully synthesized. Further delicate preparations of the porous coating of metal or alloy are planned.

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