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Improvement of hardness and fracture toughness of surface composites fabricated by high-energy electron-beam irradiation with Fe-alloy powders and VC powders

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Surface composites were fabricated by high-energy electron beam irradiation with Fe-alloy powders and VC powders, and contained a large amount (~47 vol.%) of hard Cr_2B and V_8C_7 particles without defects. According to microfracture observation, cracks were initiated at cell regions and V_8C_7 particles inside cells, and were connected with other microcracks in a zig-zag pattern, thereby showing twice the fracture toughness and hardness than the composite fabricated without VC powders. © 2009 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

Keywords: High-energy electron beam irradiation; Surface composite; Vanadium carbide; Fracture toughness

Structural materials have been increasingly exposed to severe industrial working conditions. This exposure is mostly confined to the surface region, and thus many active studies to improve surface properties have been conducted. Surface composites, in which excellent resistance to heat, corrosion and wear of carbides, borides or nitrides are fully utilized by direct irradiation from a high-energy electron beam, are good examples [1,2]. A problem in surface composites is the poor fracture toughness of the surface composite layer because it contains a number of hard particles of carbides, borides or nitrides. Since reinforcing particles work as fracture initiation sites, surface composites can be readily fractured under a low load level. Effort is now needed to determine how the fracture toughness can be improved. For example, high fracture toughness can be achieved by the development of crack-bridging zones ahead of the main propagating crack tip [3]. In addition, intensive studies have been undertaken to fabricate composites by dispersing hard particles to promote microcracking or crack bridging [4]. Deformation and fracture mechanisms related with this toughening have been investigated mainly by observations of fractured specimens after testing, but direct microfracture observation has provided little clear evidence.

In this study, the fabrication idea of surface composites containing hard carbides or borides was suggested to improve surface properties such as hardness and fracture toughness simultaneously. Three surface composites were fabricated by depositing Fe-alloy powders, together with VC powders, on the surface of a steel substrate and irradiating with a high-energy electron beam. The advantages of the VC powders were achieved by additionally precipitating vanadium carbides. Microstructure, hardness and fracture toughness of the fabricated surface composites were analyzed comparatively to understand the mechanisms of the surface property improvement.

Fe-alloy powders (ArmacorTM C+, Liquidmetal Technologies, Lake Forest, CA, USA) were used for the fabrication of surface composites. They are used commercially as thermal spray-coating powders because the coatings have excellent wear and corrosion resistance [5]. The chemical composition is Fe–30Cr–17Ni–10Co–4Mo–4B–2.5Cu–1.5Si (wt.%), and their average size is about 50 μ m. These powders were mixed with VC powders (size: 30 μ m), which have high hardness (about 2900 VHN), a high melting point (2700 °C) and a density of 5.71 g cm⁻³ [6].

After mixing Fe-alloy powders with 20–30 wt.% VC powders, the powder mixture was dried at 150 °C for 2 h. The powder mixture was placed evenly on a plain carbon steel substrate, then compacted with 120 kPa pressure (area density: 0.45 g cm⁻²). For convenience,

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Figure 1. Low-magnification optical micrograph of the V0 specimen. Nital etched.

the specimens containing Fe-alloy powders mixed with 0, 20 and 30 wt.% VC powders are referred to as V0, V2 and V3, respectively. Since the Fe-alloy powders and VC powders contained a large amount of Cr, Ni, Mo, B, Si and C, a self-fluxing effect was expected, and thus flux powders were not used [7]. A high-energy electron accelerator at the Budker Institute of Nuclear Physics, Novosibirsk, Russia was used for the irradiation [8]. The process parameters were fixed at optimal conditions (electron energy: 1.4 MeV; beam moving speed: 10 mm s⁻¹; scanning width: 50 mm; beam current: 25 mA; scanning frequency: 19 Hz; beam diameter: 11 mm).

Carbides or borides formed in the surface composites were analyzed by X-ray diffraction (XRD) and energy dispersive spectroscopy (EDS). Hardness was measured by a Vickers hardness tester under a 300 g load, and microhardness of the matrix and solidification cell region was measured under a 10 g load. A compact tension (CT)-type loading stage was installed inside a scanning electron microscope, on which a thin CT specimen with a thickness of 0.5 mm in the grooved section was placed to conduct the *in situ* scanning electron microscopy (SEM) fracture test [9]. A fatigue crack should be introduced into the CT specimen for measurements of fracture toughness, but a sharp notch with a radius of about 30-40 µm was inserted by an electric discharge machine instead, considering the difficulty in introducing a fatigue crack. It is known from the relationship between the notch tip radius and the fracture toughness in ultrahigh-strength steels and metal matrix composites that the notch toughness is somewhat smaller than or close to the fracture toughness, $K_{\rm Ic}$, when the sharp notch is sized in tens of micrometers [10]. Thus, it can be expected in the case of the surface composites that the notch fracture toughness measured with the small notch tip radius would be close to the actual value of $K_{\rm Ic}$. The load applied to the specimen was continu-



Figure 2. SEM micrographs of the (a) V0, (b) V2 and (c) V3 specimens, showing the surface composite layer. Etched by Viella's solution.

ously recorded on an X–Y recorder to obtain the load–time curve, from which the maximum load, P_{max} , was determined as P_{Q} . As the load was applied while the surface of the specimen was displayed on the screen of a scanning electron microscope, a crack was initiated and propagated from the notch tip at a certain stress intensity factor level.

Figure 1 is a low-magnification optical micrograph of the V0 specimen. It shows a smooth and glossy surface composite layer without pores and cracks as the powders and part of the substrate surface were melted and solidified. The surface composite layer/substrate interface is clearly visible, as marked by arrows. The specimen is about 1.3 mm thick, and its overall microstructure is homogeneous. The thickness of the surface composite layer is about 1.3 mm, and increases as the mixing ratio of the VC powders increases.

Figure 2(a)-(c) presents SEM micrographs of the V0, V2 and V3 specimens. The solidification cell structure is well developed in the surface composite layer, and lamellar-shaped particles are densely precipitated in the cell regions. The V2 and V3 specimens contain a number of coarse particles in the matrix inside cells (Fig. 2(b) and (c)), whereas these particles are not found in the V0 specimen (Fig. 2(a)). According to the EDS analysis results, the particles present in the cell regions and inside cells can be differentiated into vanadiumand chromium-rich particles, respectively. The volume fractions of the particles located in the cell regions or inside cells were measured, together with the cell regions and the solidification cell size, and the results are shown in Table 1. As the mixing ratio of the VC powders increases, the cell size increases but the area of the cell region decreases, thereby resulting in a decrease in the volume fraction of particles in the cell regions. As relatively coarse particles are additionally precipitated inside cells of the V2 and V3 specimens, the total volume fraction of the precipitated particles tends to increase with increasing mixing ratio of VC powders.

The phases present in the composite layers were identified by XRD analysis, as shown in Figure 3. Peaks of γ -Fe, Cr₂B and V₈C₇ are observed in the specimens, with the γ -Fe peaks being the highest. Thus, Cr₂B and V₈C₇ particles are distributed in the austenite matrix to form a cell structure. Peaks of Cr₂B are found in the V0 specimen without peaks of V₈C₇. In the V2 and V3 specimens, V₈C₇ peaks were observed, the peak height increases with increasing mixing ratio of VC powders. Thus, precipitate particles distributed in cell regions and inside cells are identified as Cr₂B and V₈C₇, respectively, as indicated by arrows in Figure 2(a)–(c).

The overall bulk hardness and microhardness of the matrix and the cell region are shown in Table 2. The overall hardness of the surface composite layer increases with increasing mixing ratio of VC powders. The hardness of the V0 specimen is 2.5 times higher than that of the carbon steel substrate (150 VHN). The microhardness of the cell regions containing Cr_2B particles is higher than that of the austenite matrix, indicating that Cr_2B particles are quite hard. The V2 specimen than the V0 specimen is harder by about 130 VHN because the matrix hardness increases as V_8C_7 particles are precipitated in the matrix. In the V3 specimen, the hardness

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