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Dislocation loop formation under various irradiations of laser and/or electron beams

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Abstract

A high-voltage electron microscope (HVEM) equipped with a laser head (laser-HVEM) was developed at Hokkaido University and is used to investigate the surface modification of semiconductors and the behaviour of lattice point defects in metals under various irradiations of laser (photon) and/or electron beams. In the present study, the annealing effect of pulsed laser irradiation on a face-centred-cubic metal was experimentally investigated and theoretically calculated. The systematic assessment of dislocation loop evolution under laser-electron sequential irradiation and laser-electron dual-beam irradiation was performed. Our results show that the rapid heating and quenching that occurred during pulsed laser irradiation caused vacancies to be introduced at the surface of the specimen and to diffuse to the interior, which led to the formation and growth of vacancy-type (V-type) dislocation loops. These loops gradually shrank and finally disappeared during the subsequent electron irradiation of the sample. During laser-electron simultaneous dual-beam irradiation, the type of loop formed, interstitial-type (I-type) or V-type loops, is determined by the relative intensities of the laser beam and electron beam, which indicates that the loop type can be controlled by changing the relative intensities of the beams. Accordingly, models of dislocation loop formation during various irradiations were proposed. The newly developed laser-HVEM instrument is expected to be employed in the exploration of mechanisms in material science, as well as in other scientific fields.

Keywords: Radiation effects; Laser-HVEM; In situ; Lattice defects; Dislocation loop

1. Introduction

Energetic beam irradiation introduces primary lattice point defects, such as interstitial and vacancy defects, in metals. These defects govern the changes in the microstructure and mechanical properties of the materials, such as the formation and growth of dislocation loops and voids [1–3], segregation near grain boundaries [4,5], precipitates [6,7], embrittlement [8,9], creep [10,11] and hardening [8,12]. Over the past a few decades, the simulation of various irradiations was achieved via the continuous development of research instruments and methods to understand the microstructural changes and the mechanisms governing these changes that occur during irradiation.

With the development of transmission electron microscopy (TEM) in the 1940s [13], it became possible to observe changes in the microstructures of materials before and after irradiation. To directly observe the changes as they occur during irradiation, which is useful for revealing the mechanisms leading to such changes, instruments and methods for in situ observation have been continuously under development, and to date, in situ techniques have been the most important and popular tools for the study of radiation effects. For example, the use of a high-voltage electron microscope (HVEM) since the 1960s [14] has made it possible to simulate the effects of neutron irradiation, except

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for collision cascades, due to the introduction of Frenkel pairs (FPs, referring to an interstitial atom and a vacancy) by electron beam irradiation [15]. Meanwhile, the combination of ion irradiation with TEM was developed for in situ observation of the simulation of high numbers of atomic displacements and collision cascades [16]. The actual environment inside nuclear reactors and in outer space is complex due to the simultaneous existence of high-energy neutrons and electromagnetic radiation [17] instead of a single energetic radiation beam. HVEM allows for the in situ observation of electron beam irradiation and utilizes a large chamber that can be conveniently connected to other instruments; since the 1970s it was coupled to an accelerator for the in situ observation of the introduction of FPs and atomic displacements resulting from multi-beam irradiation [18–24].

During heavy-ion irradiation, vacancies and interstitials are simultaneously introduced by cascade damage with a high concentration of vacancies surrounded by interstitials [25]. However, electron irradiation creates randomly distributed vacancies and interstitials (FPs). Because pulsed laser beam irradiation causes rapid heating and quenching in materials [26] and the laser head can be easily connected to other equipment, the first laser HVEM was developed in 2007 at Hokkaido University [27] to provide an environment of randomly distributed FPs with an additional source of vacancies.

Laser-HVEM has already been used in situ to observe the formation of V-type dislocation loops under irradiation with a single laser beam. Furthermore, using laser-HVEM, two new methods for measuring vacancy migration energy were also proposed [28], breaking new ground in this field after little advancement during the previous 30 years. Although the in situ observation of dislocation loop formation under single electron irradiation or single laser beam irradiation has been reported [28], the mechanisms of dislocation loop formation under laser-electron simultaneous dual-beam irradiation, a new method for studying lattice point defects, are unknown.

In the present study, both experimental studies and theoretical calculations were performed to investigate the annealing effects of pulsed laser beam irradiation on commercial SUS316L steel. Dislocation loop evolution under single laser beam irradiation, laser-electron sequential irradiation and laser-electron simultaneous dual-beam irradiation was systematically and experimentally studied. Moreover, models of dislocation loop formation under the various irradiations of laser and/or electron beams were proposed according to the results and analysis. In the future, laser-HVEM is expected to be coupled to accelerators for the in situ observation of multi-beam irradiation. This instrument can be employed not only in the evaluation of the surface modification of materials [27] and in the study of irradiation damage in metals [28] but also in broader scientific research, such as nanotechnology and the study of irradiation effects on polymer materials and foods.

2. Experimental

2.1. Material preparation

Commercial SUS316L austenite stainless steel, which is commonly employed in core internals in boiling water reactors, was used in the experimental investigations in the present study. Table 1 lists the chemical composition of the material. Sheets of the steel were mechanically thinned to a thickness of ~0.15 mm. Discs measuring 3 mm in diameter were punched from the sheets and jet-electron-milled for use as TEM specimens in the laser-HVEM irradiation experiments. The thickness of the irradiated area was determined to be 300–550 nm.

2.2. Laser-electron dual-beam irradiation

Single laser beam irradiation, laser-electron sequential irradiation and laser-electron dual-beam irradiation of the TEM specimens were performed using laser-HVEM (HVEM: Hitachi, H-1300). The laser-HVEM instrument was first described in Refs. [27,28]. A schematic of the laser-electron simultaneous dual-beam irradiation of a TEM specimen is provided in Fig. 1. The diameter of the laser beam in this study was measured to be 1.5–2 mm; the typical diameter of the electron beam for irradiation is ~2 μm. Because the diameter of the laser beam is larger than that of the electron beam, the dual-beam irradiation area is divided into three parts, which are designated as A, B and C in Fig. 1.

2.3. Experimental methods

To investigate the annealing effects of the pulsed laser beam (Nd:YAG laser, Inlite II, Continuum) irradiation of the material, a bulk sample of SUS316L was first irradiated with a single laser beam in air at room temperature for 15,000 pulses with an energy density of 124 mJ cm^{-2}. The central wavelength was 532 nm, and the pulse repetition rate was 2 Hz. After laser irradiation, the surface of the sample was studied by scanning electron microscopy

Table 1
Chemical composition of present SUS316L steel (mass%).

C	Si	Mn	P	S	Ni	Cr	Mo	V	Al	N
0.013	0.20	1.28	0.024	0.0010	13.32	17.24	2.04	0.04	0.014	0.0396