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## Deformation Twinning in Pure Nickel Induced by a High-Current Pulsed Electron Beam

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**Abstract** Recent experimental evidence has shown that face-centered-cubic materials that are not normally associated with deformation twinning will twin during moderate high-current pulsed electron beam (HCPEB) processing. In this paper, we present an experimental investigation of deformation twinning in pure multi-crystalline nickel exposed to HCPEB irradiation from a Nadezhda-2 HCPEB device. The samples, which were irradiated with various numbers of pulses, were examined. The formation of a large number of twin lamellae on the treated surface was observed in the regions without surface melting. Moreover, a large strain located at the twin lamellae deformation was found. Transmission electron microscope analysis revealed that the deformation twinning was indeed triggered during the HCPEB irradiation. This suggests that the very high stresses and superfast strain rates induced via the rapid heating and cooling caused by the HCPEB irradiation lead to deformation twinning in coarse-grained nickel.

**Keywords** High-current pulsed electron beams (HCPEB) · Pure nickel · Deformation · Twin

### الخلاصة

لقد أظهرت الدلائل التجريبية الحديثة أن مواد fcc الزوجية التي لا تصاحب طبيعياً تشوه التوأمة سوف تنتج توأما باستخدام معالجات إشعاعات تيارات إلكترونية نابضة HCPEB بدرجة متوسطة المرتفع. في هذه الورقة العلمية نقدم بحثاً تجريبياً لتشوه التوأمة في النيكل النقي متعدد البلورة تحت إشعاعات HCPEB باستخدام مصدر HCPEB. وقد تم فحص العينات تحت الإشعاع باستخدام أرقام نوابض عديدة. ووجد أنه يمكن ملاحظة تكون عدد كبير من صفحات التوائم فوق السطح المعالج بدون صهر السطح مع إمكانية تحديد وقوع التواء في تشوه صفحات التوائم. وكشفت نتائج مجهرية الانتقال الإلكتروني (TEM) أن تشوه التوأمة أثير في الحقيقة خلال شوط إشعاع HCPEB. ويمكن الاقتراح أن توترا بغير مرتفعة مع سرعة التواء فائقة السرعة المستحثة بتسخين و تبريد سريعين نتيجة لإشعاعات HCPEB هو الذي يسبب تشوه التوأمة في حبيبات النيكل الخشنة.

### 1 Introduction

The high-speed deformation of metals has attracted interest from many fields in science and technology. A major focus of recent research has been the deformation mode of ductile materials undergoing high-speed deformation, which may differ from that in normal-speed deformation. Generally, the response of metals to

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mechanical stress can produce two types of plastic deformation: dislocation slipping and deformation twinning [1]. Dislocation slipping has received the most attention from both theoretical and experimental researchers during the past 60 years. Deformation twinning also constitutes a significant number of the modes of deformation and can dominate plastic deformation for specific deformation conditions. In body-centered-cubic (bcc) metals, deformation twinning occurs at a low temperature, where it becomes more favorable than dislocation slip processes [1]. Deformation twinning is rare in face-centered-cubic (fcc) metals. It normally requires low temperatures and high stresses or strain rates, but some fcc alloys with low stacking fault energies twin more easily [2]. Of the fcc metals, pure aluminum has been traditionally identified as an example of a metal that does not exhibit deformation twinning because its stacking fault energy is too high [2]. For pure nickel, which has an fcc structure and a moderate stacking fault energy ( $0.25 \text{ J/m}^2$ ), experimental evidence of deformation twinning is also very rare (as far as we know), especially for coarse-grained nickel. However, more recently, our experimental work has indicated that deformation twinning can occur in single-crystal aluminum during high-current pulsed electron beam (HCPEB) irradiation [3].

During the past decade, HCPEBs, which were initially developed at the Tomsk Institute of High Current Electronics [4], have attracted a great deal of attention in the field of material surface modifications. HCPEBs are high density energy sources that allow for the deposition of energy over a short period in small depths near the material surface. Such pulsed electron irradiation induces (i) rapid heating and cooling in the surface [5] and (ii) the formation of dynamic stress fields [6]. The combination of these processes makes it possible to substantially modify the surface characteristics [7, 8] and, in many cases, improve the mechanical properties of the material faster and more efficiently than conventional surface treatment techniques [9, 10]. However, our previous studies have indicated that HCPEBs serve as an effective platform for the investigation of the microstructures and mechanisms of high-speed deformation. The characterization of deformation microstructures is very important for the development of our understanding of thermo-mechanical evolution during high-speed deformation.

In this paper, we investigate the microstructure response of the near-surface layer of pure nickel exposed to HCPEB irradiation. We used a HCPEB source to irradiate the pure bulk nickel. We present an experimental investigation of the deformation microstructures of pure multi-crystalline nickel. We observed that twinning deformation in the surface layer of nickel was triggered after HCPEB irradiation. This study focuses on the formation of deformation twins and the interaction mechanisms between HCPEB irradiation and materials.

## 2 Material and Experimental Procedure

A pure bulk nickel sample ( $10 \times 10 \times 8 \text{ mm}^3$ ) was subjected to HCPEB irradiation. Prior to HCPEB treatment, the pure nickel samples were annealed at 973 K for 2 h in a vacuum tube furnace to reduce the effect of rolling and mechanical processing. Also, all the samples surfaces were ground and polished.

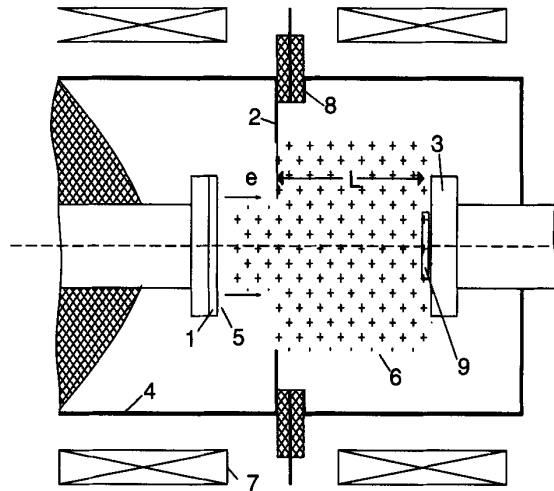
A schematic of the HCPEB source (Nadezhda-2) is shown in Fig. 1. The HCPEB source can produce electron beams with the following characteristics: electron energy 10–40 keV, pulse duration 0.5–5  $\mu\text{s}$ , peak current density 0.5–5  $\text{J/cm}^2$  and cross-section area 10–50  $\text{cm}^2$ . The accelerating voltage, magnetic-field strength and the anode–collector distance can be used to control the beam energy density.

In this study, the pure nickel samples were irradiated using working parameters of: accelerating voltage 21.6 kV, number of pulses (1, 5 and 10), pulse duration 1  $\mu\text{s}$  and anode–target distance 8 cm.

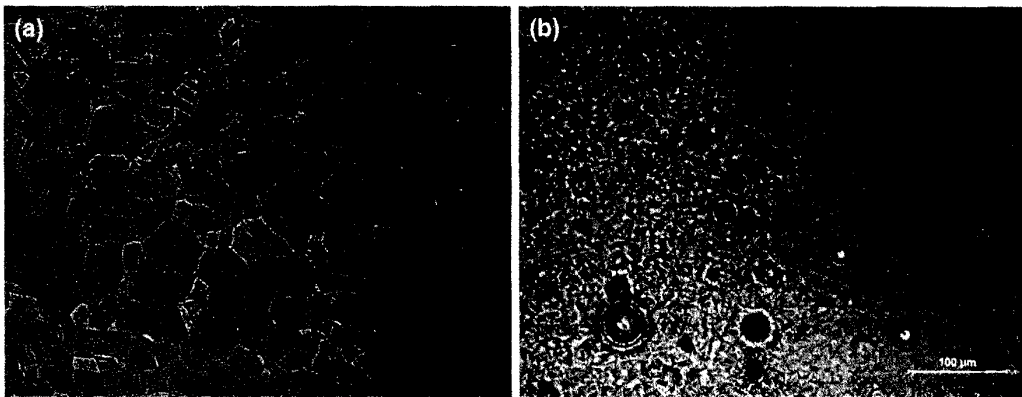
The microstructure changes in the deformed layer of the nickel samples were characterized using a LEICA DM-2500M optical microscope (Wetzlar, Germany), JEOL JSM-7001F scanning electron microscopy (SEM, Tokyo, Japan) and JEM-2100F (JEOL) transmission electron microscope (TEM). The foils used in the TEM observations were obtained by preparing a one-sided mechanically prethinned, dimpled and electrolytical thinned plate. The plates were thinned until electron transparency occurred. The depth of the near-surface layer that was analyzed was between 0.5 and 1.5  $\mu\text{m}$ .

## 3 Experimental Results

Figure 2 shows the surface morphology of the nickel samples in their initial state and after HCPEB irradiation. For the initial sample, chemical etching in nitrohydrochloric acid was conducted on the mechanically polished surface prior to characterization. From Fig. 2a, it can be seen that the microstructure is composed of large grains with sizes ranging from approximately 10 to 30  $\mu\text{m}$ . Fewer annealed twins were observed in some



**Fig. 1** Schematic of the HCPEB source using plasma-filled systems based on vacuum spark plasma. 1 Cathode, 2 anode, 3 collector, 4 vacuum-chamber, 5 cathode plasma, 6 anode plasma, 7 solenoid, 8 spark source and 9 sample



**Fig. 2** Surface metallographic images of nickel samples. a Initial state and b after the ten-pulse HCPEB treatment with an accelerating voltage of 21.6 kV

grains. For the irradiated samples, the microstructures on the irradiated surface could be observed directly and the chemical etching process was not needed. Figure 2b shows the sample, which was irradiated with ten pulses from a beam with an accelerating voltage of 21.6 kV. Craters were the most common feature on the surface, which is typical of many HCPEB irradiated metal surfaces and has been observed by many researchers [4, 10]. From previous studies, such a morphology is the result of local sublayer melting and eruption through the solid outer surface [10].

Figure 3 shows SEM images of the regions irradiated with ten pulses from a beam with an accelerating voltage of 21.6 kV. It can be seen that severe deformations in the surface layer occurred after HCPEB irradiation. Many markings of deformation are clearly visible on the irradiated surface. Dense deformation bands were present in almost all the grains, and these bands never cross the grain boundary. In some cases, groups of intersecting bands can be seen in the grains. Note that many more twin lamellae features are present in this sample compared with the original sample. This suggests that additional twin structures were introduced on the irradiated surface after HCPEB treatment.

In Fig. 3, a displacement of the deformation bands can be seen. This results in zigzag-shaped slip bands where the twin lamellae intersect, which is indicated by the arrows in Fig. 3. These displacements were induced by the twin lamellae. Therefore, the deformation bands probably formed first. Then, the intersecting twin lamellae formed. This order of formation is likely because the latter are straight and the former are zigzag-like. This result suggests that the twin lamellae, which are shown in Fig. 3, formed after dislocation



**Fig. 3** SEM images showing the surface morphology of the pure nickel samples after the ten-pulse HCPEB irradiation with an accelerating voltage of 21.6 kV

slipping during HCPEB treatment. Therefore, the twin lamellae that formed after HCPEB irradiation are the result of deformation caused by the very high strain rate induced by HCPEB irradiation. This also indicates that the deformation is concentrated at the twin lamellae. From the displacement of the deformation bands between the upside and underside of the twin lamellae, we determined that a larger strain was located in the twin lamellae.

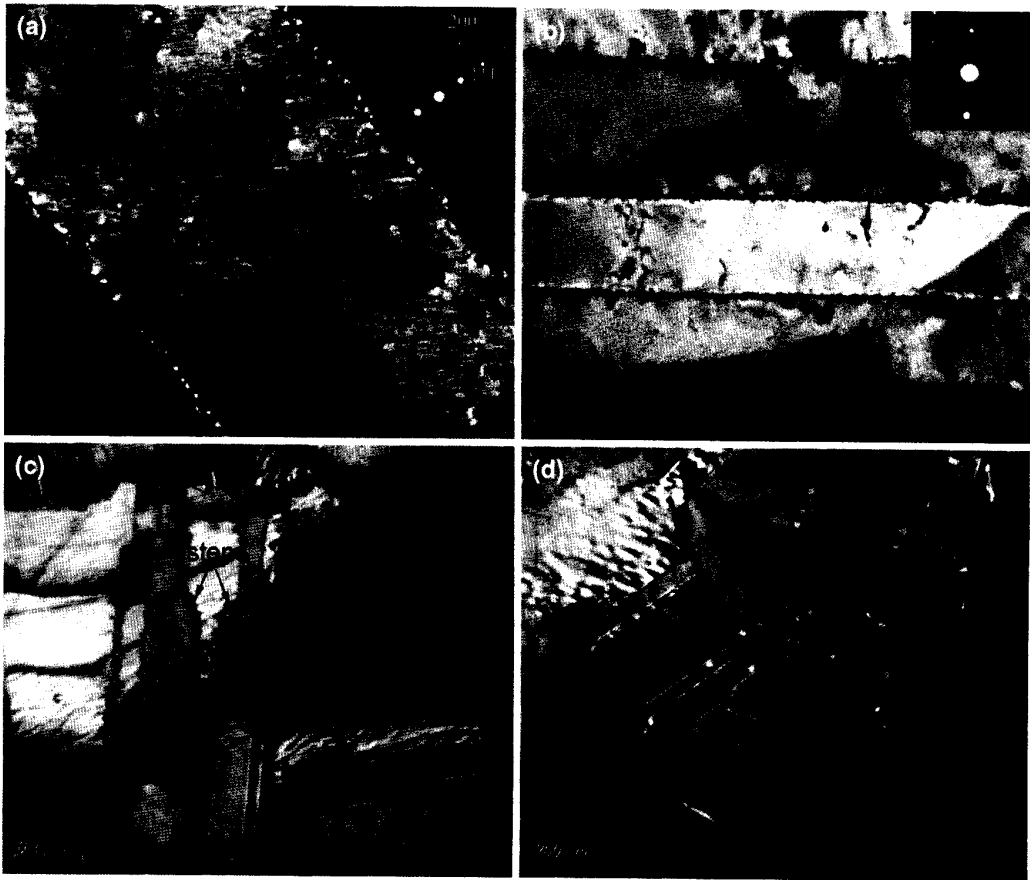
To obtain direct crystallographic evidence, the thin foils for TEM observation were prepared using mechanical pre-thinning, dimpling and jet electrolytical thinning from the substrate side. Figure 4a shows a TEM image of twin lamellae and the corresponding selected-area electron diffraction (SAED) pattern (insert) after one pulse HCPEB irradiation. It can be seen that there are dense dislocation walls (DDWs) that are about 10 nm wide throughout the whole twin lamellae structure. These DDWs are also frequently seen inside many grains. This suggests that the DDWs formed simultaneously inside the twin lamellae and within the grains because the initial sample has no DDW microstructures. Therefore, the twin lamella that is shown in Fig. 4a is probably an annealed twin lamella because all the twin lamellae that were initially annealed exhibit DDWs after HCPEB irradiation.

In addition to the annealed twins, we found bundles of twin lamellae with no dislocations or other dislocation configurations. This can be seen in Fig. 4b. A SAED pattern taken along a [011] zone axis of the twin bundles (inset of Fig. 4b) shows the superposition of a couple of {011} diffraction patterns. These patterns are symmetrical to each other with respect to the {111} plane, which is indicative of a twin relationship ({111}/[112]) type among the lamellae. Moreover, these stepped twin boundaries are frequently seen in these twin bundles (Fig. 4c). This marking is typical of deformation twinning [11]. These results indicate that these twin bundles are deformation twins that are induced by HCPEB irradiation because no such twin bundles were present in the initial samples. Furthermore, the fact that there are no DDWs inside the twin bundles also suggests that they are deformation twins. Note that the number of deformation twins increases with the number of irradiation pulses for the samples in this study. This can be seen in Fig. 4d.

Figure 5 shows TEM images of the micro-twins and their corresponding SAED patterns in deformed grains for five HCPEB pulses. This further confirms that deformation twinning is induced by the HCPEB irradiation.

#### 4 Discussion

Dislocation slipping and twinning are the two primary deformation mechanisms. Moreover, they are competing processes. Dislocations are thermally activated processes. Various dislocation configurations, including dislocation cells, walls, geometrically necessary boundaries and incidental dislocation boundaries, can be formed via the manipulation and rearrangement of dislocations when a polycrystalline nickel is deformed at temperatures at or above room temperature [12]. When nickel is deformed at a high strain rate, however, dislocation manipulation and rearrangement are suppressed, which leads to the formation of poorly developed dislocation cells and a small number of microbands (Fig. 4a). Another consequence of the suppressed dislocation process



**Fig. 4** Bright-field TEM images of the twin lamellae and their corresponding SAED patterns. **a** 1 pulse, **b** 5 pulses, **c** and **d** 10 pulses



**Fig. 5** TEM image of the micro-twins and the corresponding SAED pattern after a five-pulse HCPEB irradiation



stress ( $P$ ) in the surface layer (according to a numerical simulation developed by Zou et al. [15]) can be as high as  $10^3$  MPa, and the estimated deformation rate can be as high as  $10^4$ – $10^5$  s<sup>-1</sup> in aluminum. Stress and deformation rates of this level can easily induce violent deformation in metallic materials. For nickel, which has a higher yield intensity than aluminum, HCPEB should induce higher stress values. This may be the real reason for the formation of the deformation twins in nickel.

## 5 Conclusions

Superfast deformation provides a means to access atomic interactions in materials and probe the details of bonding and defect physics. Thus, understanding the response of matter to rapid loading is an inherently multi-disciplinary challenge. This is also true because of the interplay of deformations and the short time scales over which they occur. In this paper, we investigated the microstructure changes that occur on HCPEB irradiated surfaces of pure nickel. A large number of twin lamellae on the treated surface were observed in the regions without surface melting and with large strains. Our TEM analysis demonstrated that deformation twinning is triggered by HCPEB irradiation. Our results also suggest that very high stresses and strain rates are induced by the rapid heating and cooling caused by HCPEB irradiation. These high stresses and strain rates could cause deformation twinning in coarse-grained nickel.

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