

(12) **United States Patent**
Mardilovich et al.

(10) **Patent No.:** **US 9,410,260 B2**
(45) **Date of Patent:** **Aug. 9, 2016**

(54) **METHOD OF FORMING A NANO-STRUCTURE**
(75) Inventors: **Peter Mardilovich**, Corvallis, OR (US);
Qingqiao Wei, Corvallis, OR (US);
Anthony M. Fuller, Corvallis, OR (US)

(73) Assignee: **Hewlett-Packard Development Company, L.P.**, Houston, TX (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 508 days.

(21) Appl. No.: **13/822,062**

(22) PCT Filed: **Oct. 21, 2010**

(86) PCT No.: **PCT/US2010/053588**

§ 371 (c)(1),
(2), (4) Date: **Mar. 11, 2013**

(87) PCT Pub. No.: **WO2012/054045**

PCT Pub. Date: **Apr. 26, 2012**

(65) **Prior Publication Data**

US 2013/0175177 A1 Jul. 11, 2013

(51) **Int. Cl.**
C25D 11/12 (2006.01)
C25D 11/34 (2006.01)
(Continued)

(52) **U.S. Cl.**
CPC **C25D 5/022** (2013.01); **B81C 1/00031**
(2013.01); **C25D 11/045** (2013.01);
(Continued)

(58) **Field of Classification Search**
CPC C25D 5/022; C25D 11/12; C25D 11/045;
C25D 11/34; B81C 1/00031
USPC 205/136
See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

5,581,091 A 12/1996 Moskovits et al.
6,359,288 B1 3/2002 Ying et al.

(Continued)

FOREIGN PATENT DOCUMENTS

CA 2137013 11/1994
CN 101255588 9/2008

(Continued)

OTHER PUBLICATIONS

Takahashi, H., et al., "Anodic film growth on Al layers and Ta—Al metal bilayers in citric acid electrolytes", *Electrochimica Acta*, 2005, p. 5065-5075.*

(Continued)

Primary Examiner — Keith Hendricks
Assistant Examiner — Stefanie S Wittenberg
(74) *Attorney, Agent, or Firm* — Dierker & Kavanaugh, P.C.

(57) **ABSTRACT**

A method of forming a nano-structure involves forming a multi-layered structure including an oxidizable material layer established on a substrate, and another oxidizable material layer established on the oxidizable material layer. The oxidizable material layer is an oxidizable material having an expansion coefficient, during oxidation, that is more than 1. Anodizing the other oxidizable material layer forms a porous anodic structure, and anodizing the oxidizable material layer forms a dense oxidized layer and nano-pillars which grow through the porous anodic structure into pores thereof. The porous structure is selectively removed to expose the nano-pillars. A surface (I) between the dense oxidized layer and a remaining portion of the oxidizable material layer is anodized to consume a substantially cone-shaped portion of the nano-pillars to form cylindrical nano-pillars.

12 Claims, 12 Drawing Sheets

