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Josell et al.

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(54) **SUPERCONFORMAL FILLING COMPOSITION AND SUPERCONFORMALLY FILLING A RECESSED FEATURE OF AN ARTICLE**

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CPC C25D 3/48; C25D 3/362; C25D 3/62; C25D 7/00; C25D 7/123; C25D 5/02;
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(56) **References Cited**

U.S. PATENT DOCUMENTS

2004/0065225 A1* 4/2004 Ruebel C25D 3/62 106/1.18
2005/0092616 A1* 5/2005 Hu C25D 3/02 205/266

* cited by examiner

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(57) **ABSTRACT**

Superconformally filling a recessed feature includes: contacting the recessed feature with superconformal filling composition that includes: $Au(SO_3)_2^{3-}$ anions; SO_3^{2-} anions; and Bi^{3+} cations; convectively transporting $Au(SO_3)_2^{3-}$ and Bi^{3+} to the bottom member of the recessed feature; subjecting the recessed feature to an electrical current to superconformally deposit gold from the $Au(SO_3)_2^{3-}$ on the bottom member relative to the sidewall and the field, the electrical current providing a cathodic voltage; and increasing the electrical current subjected to the field and the recessed feature to maintain the cathodic voltage between −0.85 V and −1.00 V relative to the SSE during superconformally depositing gold on the substrate to superconformally fill the recessed feature of the article with gold as a superconformal filling of gold, the superconformal filling being void-free and seam-free.

27 Claims, 56 Drawing Sheets