# Thursday Afternoon Poster Sessions, April 27, 2017

### New Horizons in Coatings and Thin Films Room Grand Exhibit Hall - Session FP

#### Symposium F Poster Session

# FP-3 Monolayer Controlled Deposition of ZnO Thin Films by Catalytic Reaction-assisted Chemical Vapor Deposition, S Ono, T Saitou, R Tajima, Y Tamayama, Kanji Yasui, Nagaoka University of Technology, Japan

The large bandgap (3.37 eV at RT) and exciton binding energy (60 meV) of ZnO [1] have recently stimulated intensive research into optoelectronic device applications, such as light-emitting diodes and laser diodes that operate in the ultraviolet region [2-6]. We have previously developed a new CVD method for ZnO film growth based on the reaction between alkylzinc (DMZn) and high-temperature H<sub>2</sub>O generated by a Pt-catalyzed exothermic H<sub>2</sub>-O<sub>2</sub> reaction. The resulting ZnO films grown on a-plane (11-20) sapphire (a-Al<sub>2</sub>O<sub>3</sub>) substrates exhibited excellent optical and electronic properties [7]. Superlattices are often used to enhance the efficiency of optical emission devices. Such structures involve alternating layers with different compositions, with each layer often being just a few molecules thick. It is therefore necessary to be able to control the growth process at the monolayer level. Therefore, in the present study, the applicability of catalytic reaction-assisted CVD using a pulsed gas supply was investigated for monolayer controlled growth of ZnO on a-Al<sub>2</sub>O<sub>3</sub> substrates.

Except for the gas supply mode, the CVD apparatus and growth procedure were the same as those previously reported [7]. DMZn gas was supplied to the reaction zone in pulses with durations of 4 and 8 ms, and was reacted with a catalytically generated high-energy  $H_2O$  beam effused from a de Laval nozzle. The growth rate per pulse was evaluated based on the final film thickness and the total number of DMZn pulses. Pulse rates of 2 and 4 Hz were used, with the gas being supplied for 30 and 15 min, respectively. For a pulse duration of 8 ms, based on the final ZnO film thicknesses of 1300 and 1400 nm, the deposition rate was 0.36 and 0.39 nm per pulse for a pulse rate of 2 and 4 Hz, respectively. For a pulse duration of 4 ms, based on the final film thickness of 1400 nm, the deposition rate was 0.19 nm per pulse. Since the c-lattice constant of ZnO is 0.52 nm, the thickness of a single molecular layer is 0.26 nm. Therefore, from the above results, the thickness of the deposited layer could be controlled to less than the thickness of a single molecular layer using catalytic reaction-assisted CVD with a 4 ms pulsed gas supply.

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FP-6 Optical and Electronic Properties of MoS2: Joint Theoretical/Experimental Study, Miller Eaton, H Sirikumara, H Samassekou, D Mazumdar, Southern Illinois University, USA; L Liyanage, M Nardelli, University of North Texas, USA; T Jayasekera, Southern Illinois University, USA

First-principles calculations of the optoelectronic properties of semiconductors using density function theory (DFT) have suffered from slight inaccuracies due to the exchange energy interaction approximations used. The inclusion of the Hubbard parameter U allows for more accurate predictions with a negligible increase in computational time. We use the DFT + U approach to perform ab initio calculations of the optical and electronic properties of thin film MoS2. We then fabricate pristine MoS2 thin films on BN-buffered silicon substrates via magnetron sputtering. Fewlayer films are first confirmed with X-ray reflectivity and diffraction measurements, and then further characterized using variable angle spectroscopic ellipsometry (VASE). Agreement of DFT + U predictions with layer specific modeling of VASE data demonstrates the viability of sputtering for large-area growth. This study paves the way for the controlled introduction of chemical dopants to further improve electronic and optoelectronic properties of MoS2 for nanoscale device application.

FP-7 Possibility of Selective and Morphology-Controlled Growth of CuO and Cu<sub>2</sub>O Films, *Tomoaki Terasako*, *K* Ohnishi, *H* Okada, *S* Obara, Ehime University, Japan; *M* Yagi, National Institute of Technology, Kagawa College, Japan

Both CuO (Eg=~1.35 eV) and Cu<sub>2</sub>O (Eg=~2.1 eV) exhibit p-type conduction. These materials have attracted much attention because of their promising

applications, such as photocatalytic water splitting, solar cells, electrode materials for lithium-ion batteries, gas-sensing devices and so on. Chemical bath deposition (CBD) is performed at temperatures typically lower than 100 °C, which allows us to use polymers as substrate materials. In this paper, we will discuss the possibility of the selective and morphology-controlled growth of CuO and Cu<sub>2</sub>O films on Au/SiO<sub>2</sub>/Si(100) substrates by CBD from the same precursor, *i.e.* Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O (CuNit).

The CuO films grown from the aqueous solutions of CuNit with the different pH values adjusted by the use of NH<sub>3</sub>(aq) were composed of the needle-like grains whose average length increased with increasing pH value. At pH≥10.5, the needle-like grains were organized in the caddis clew-like shapes. The most probable mechanism for assembling the needle-like grains is the "oriented attachment" in which the rotations of the needle-like grains can be driven by torques introduced by the misalignment of the neighboring grains [1, 2]. The appearance of the caddis clew-like shapes is probably due to the imperfect rotation of the needle-like grains caused by the increase in the average length.

The Cu<sub>2</sub>O films were grown from the mixed aqueous solutions of CuNit and C<sub>6</sub>H<sub>12</sub>N<sub>4</sub> (HMT) with the assistance of a piece of Fe plate immersed in the solution, denoted by "Fe-assisted CBD". No Cu<sub>2</sub>O film could be obtained without the immersion of the Fe plate [3]. The reduction from the  $Cu^{2+}$  ions to the Cu<sup>+</sup> ions is required for the formation of Cu<sub>2</sub>O from CuNit. Oxidationreduction potential (ORP) measurement during the Fe-assisted CBD process revealed that the ORP exhibited a sudden decrease just after immersing the Fe plate. Moreover, the ORP values during the Fe-assisted CBD were overall low in comparison with those during the usual CBD process. Taking into account the fact that  $\alpha$ - and  $\delta$ -FeOOH were formed on the surface of the Fe plate during the CBD process, the CBD solution in which the Fe plate was immersed had high concentration of electrons contributing to the reduction of Cu2+ ions. The preferential growth direction of the Cu2O film changed from [111] to [100] with increasing CuNit (or HMT) concentration, accompanied with the change from the triangular-plate grains to the foursided pyramidal grains.

This work was supported by JSPS KAKENHI Grant Number JP26390029.

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FP-8 Thermal Stability of Arc Evaporated Oxide, Nitride, Oxinitride, and Oxide/Nitride Coatings within the Systems Al-Cr-N and Al-Cr-O, Robert Raab, CDL-AOS TU Wien, Austria; C Koller, TU Wien, Austria; S Kolozsvári, Plansee Composite Materials GmbH, Germany; J Ramm, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein; P Mayrhofer, TU Wien, Austria

The most common materials systems synthesised by cathodic arc evaporation for industrial applications are nitrides or oxides (e.g., TiAlN, CrAlN,Al<sub>2</sub>O<sub>3</sub>,...). Among these, Al-Cr-based films find application in multitude of applications, such as forming or cutting tools, which not only required a consistent performance over a temperature range up to 1000°C and beyond, but also the capability to withstand oxidising environments.

In this regard, we have studied the thermal stability of Al<sub>x</sub>Cr<sub>1-x</sub>N/(Al<sub>x</sub>Cr<sub>1-x</sub>)<sub>2</sub>O<sub>3</sub> multilayer coatings and compared them with the homogenously grown counterparts Al<sub>x</sub>Cr<sub>1-x</sub>N, (Al<sub>x</sub>Cr<sub>1-x</sub>)<sub>2</sub>O<sub>3</sub>, as well as its combination in terms of quaternary Al-Cr-O-N.

We used a combination of differential scanning calorimetry (DSC), thermal gravimetric analysis (TGA), X-ray powder diffraction (XRD), and hardness investigations after annealing the samples up to 1500 °C in vacuum for one hour to investigate the decomposition of the Al-Cr-based oxide, nitride, oxinitride, and oxide/nitride coatings.

Based on our results we can conclude that a multi-layer arrangement with optimized bilayer period is superior to the monolithically grown  $Al_xCr_{1-x}N$ ,  $(Al_xCr_{1-x})_2O_3$ , and quaternary Al-Cr-O-N.

**FP-9** Parametric Study of TiN Thin Films Deposited on 316 L Substrates by HiPIMS, *L Melo-Máximo*, ITESM-CEM, Mexico; *F Estrada-Martinez*, Termoinnova S.A. de C.V., Mexico; *D Melo-Maximo*, TRAMES S.A. de C.V., Mexico; *Joaquin Oseguera*, ITESM-CEM, Mexico

Thin films of TiN were prepared by High-power impulse magnetron sputtering (HiPIMS) on 316L substrates. The main objective of this work was to evaluate the thin film growth under various conditions, in order to show the effect that process parameters have on the properties of the films. The microstructure and the adhesion of the coatings were studied by

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the characterization with scanning electron microscopy, optical microscopy, x-ray diffraction and scratch test.

**FP-11 Transition Metal Dichalcogenides for Next Generation Semiconductor Devices**, *B Sirota*, University of North Texas, USA, U; *A Waite*, *N Glavin*, Air Force Research Laboratory, USA; *C Muratore*, University of Dayton, USA; *S Krylyuk*, *A Davydov*, National Institute of Standards and Technology, USA; *Andrey Voevodin*, University of North Texas, USA

New advances in technology encourage the proliferation of next generation electronic devices. Two dimensional semiconductor materials such as MoS<sub>2</sub>, WS<sub>2</sub>, MoTe<sub>2</sub>, MoSe<sub>2</sub> and similar compounds have gained significant interest for their capabilities due to their size, flexibility, optical transparency, and tunable electronic bandgap. In addition, ultra-thin amorphous BN has gained recent interest as a dielectric material due to its large band gap, flexibility, optical transparency, and low temperature processing compatible with polymer substrates. Herein, we present a study on 2D transition metal dichalcogenides prepared by physical vapor deposition (MoS<sub>2</sub>) and mechanical exfoliation (MoTe<sub>2</sub>). We also examine their heterostructures with pulsed laser deposited ultra-thin amorphous BN layer, and integrated into electronic devices. Proof-of-concept field effect transistors with 2D semiconductors serving as the channel material are made using conventional electronic beam lithography and lift off. Microstructure, electronic and optical properties are determined and compared to devices made from traditional techniques in the recent literature. These results demonstrate the functionality of 2D transition metal dichalcogenide layers and their heterostructures with amorphous ultra-thin BN as a use for electronic devices with advanced functionality.

FP-13 Microscopic Barrier Mechanisms and Interface Damage Behavior of Two-dimensional Nanomaterials, Pu Jibin, L Wang, J Xue, Key Laboratory of Marine New Materials and Related Technology, Ningbo Institute of Material Technology and Engineering, Chinese Academy of Sciences, China The serious surface damage of high-tech facilities in the marine and spatial environment has become a key problem that restrains the reliability and life span of marine engineering facilities and spacecrafts. The common mechanism of material damage in the marine and spatial environment is infiltrated and eroded by the medium of external environment. Twodimensional nanomaterials (graphene, hexagonal boron nitride, disulfide, etc.) have a wide coverage of surface area and excellent mechanical, electrical, thermal and obstructing properties. So they have natural advantages as materials of resisting ion penetration and atomic oxygen erosion in the marine and spatial environment. By studying the influence of the electronic structure, electric conductivity, thermal conductivity, layer number, surface functional group and defect degree of two-dimensional nanomaterials on obstructing marine medium and atomic oxygen and the influence of the controllable dispersion, ratio, distribution pattern and multi-scale interface of two-dimensional nanomaterials on the damage of composite coatings, this paper expounds the obstructing regulation mechanism of two-dimensional nanomaterials so as to provide theoretical guidance for developing new-type long-life materials of marine protection and spatial anti-atomic oxygen.

**FP-14 Corrosion Performance of Waterborne Epoxy Coating using Noncovalent Dispersion of Graphene as Inhibitor**, *Shuan Liu*, *H Zhao*, Key Laboratory of Marine Materials and Related Technologies, Zhejiang Key Laboratory of Marine Materials and Protective Technologies, Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, China; *P Jibin*, Key Laboratory of Marine Materials and Related Technologies, Zhejiang Key Laboratory of Marine Materials and Related Technologies, Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, Ningbo 315201, P. R. China, China; *L Wang*, Key Laboratory of Marine Materials and Protective Technologies, Ningbo Institute of Materials and Protective Technologies, Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, China

To improve the anticorrosion performance of waterborne epoxy coating and expand its practical applications, Graphene was initially stabilized by sulfonated polyaniline in aqueous solution, and its structure and dispersant performance were investigated by SEM.TEM Raman and AFM respectively. Then the corrosion behaviour of different composite coatings in 3.5% NaCl solution was studied based on a series of electrochemical measurements. Results indicated that graphene could be dispersed uniformly in water. Sulfonated polyaniline was not only a graphene dispersant but also an corrosion inhibitor. The addition of well-dispersed graphene into waterborne epoxy coating improved corrosion protection significantly compared with neat waterborne epoxy coating.

#### FP-16 Production and Testing of Enhanced Photocatalytic Coatings onto Nanoparticles by Magnetron Sputtering, *P Kelly, Marina Ratova, G West,* Manchester Metropolitan University, UK

Magnetron sputtering is a well-established technique for the deposition of high quality metallic and ceramic coatings onto a wide range of substrate materials and forms. It is not generally suitable, though, for the coating of fine particulates (particle sizes from 10s of nm to 100s of  $\mu$  m). This paper, however, describes the use of an oscillating mechanism to manipulate particulates under two co-planar magnetrons, such that uniform coverage of the particles is achieved.

This technique has been used to deposit a range of coatings onto photocatalytic anatase titania nanoparticles (PC500), with the aim of overcoming some of the limitations of this material, specifically rapid charge carrier recombination and low visible light activity. The materials selected include bismuth tungstate, a visible light active photocatalyst, which can increase charge carrier numbers and lifetimes through semiconductor coupling with titania; and carbon, which can be used as a dopant to increase overall activity. The bismuth tungstate coatings were deposited by reactive magnetron co-sputtering from two targets. Two strategies were used for carbon doping; direct sputtering from a carbon target and reactive sputtering using CO<sub>2</sub> as a process gas.

The nanoparticles were characterised by SEM, EDX, TEM, XRD and BET surface area measurements. Photocatalytic activity was assessed using an acetone degradation test under visible light irradiation, in which the rate of  $CO_2$  evolution was measured as a function of the surface area of the sample. Preliminary results indicate that the bismuth tungstate coatings, where the Bi/W content is in the ratio of 2:1 can significantly increase the visible light activity of the titania product. The mechanism through which this enhancement occurs is discussed in terms of more efficient separation of the charge carriers and the inherent activity of the coating.

#### FP-17 3D Printing of Metal Oxide Semiconductor?, Chuong Nguyen, University of Auckland, New Zealand; J Leveneur, GNS Science, New Zealand; M Taylor, J Metson, University of Auckland, New Zealand

Ion-induced reduction can potentially provide two important features for metal oxide semiconductors: amorphous state and tuneable contact resistance. It is well known that ion bombardment causes amorphitization of target materials. Ion fluence can also be controlled to reduce oxide to metallic state in various proportions, tuning the metal – semiconductor contacts from Schottky to Ohmic.

This study involved argon ion bombardment on multi-valent iron oxides. The reduction extents, in terms of oxidation state and effective depths, as a function of ion fluence is characterized by energy-resolved x-ray photoelectron spectroscopy (ERXPS), complemented by x-ray absorption of near-edge structure (XANES). The correlation is assessed against the transport of ions (TRIM) simulation. Our early results indicate a possible method to build up a functionally graded thin-film transistor layer by layer using physical vapor deposition (PVD) coupled with a supplementary ion gun.

FP-18 A Proposal for Laser Annealing Process with Continuous Wave Nd:YAG Laser ( $\lambda_o$  = 532 nm) for Photovoltaic CIGS Thin Films: Effect of Laser Annealing Time on Optical and Electrical Properties, *MyoungHan Yoo*, *D Lee*, Chosun University, Republic of Korea; *Y Jun*, Hyobjin Jeongbo Co., Inc., Republic of Korea; *P Ko*, *N Kim*, Chosun University, Republic of Korea

Culn1-xGaxSe2 (CIGS) thin-film solar cells are strongly considered as an absorber layer in light-weight and flexible thin-film solar cells . For preparing CIGS thin film at low cost, critical problems still remained in the selenization process of the sputtered Cu-In-Ga precursors by using H<sub>2</sub>Se vapor, such as highly toxic H<sub>2</sub>Se, poor adhesion to the back contact and the slow rate of reaction. A non-selenization process was proposed to be developed for fabricating CIGS thin film by these authors; however, the annealing temperature was over the decomposition temperature of the typical flexible polymer substrates. A novel method by using a rapid laser annealing process has also been proposed and investigated to demonstrate the feasibility of crystallization and grain growth for CIGS thin film. In our prior study, a continuous wave (CW) second harmonic wavelength of Nd:YAG ( $\lambda_o$  = 532 nm) laser was selected to improve the microstructure of CIGS thin film with a change of the laser optical power from 2.25 to 3.00 W for 200 sec. CIGS chalcopyrite (112), (220/204) and (312/116) phases were formed in the laser-annealed CIGS thin films, but the weak diffraction

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peaks of Cu-Se related phases were also observed due to the insufficient energy for the grain growth. In this study, the laser annealing time was controlled to accomplish long-range atomic diffusion, chemical reactions and grain growth with a considering mass-production manufacturing. The crystal structures and chemical compositions of the laser-annealed thin films were analyzed by X-ray diffraction and energy-dispersive X-ray spectroscopy as a function of laser annealing time. The effects of them on the optical properties and electrical characteristics of CIGS thin films were examined by using an UV-Visible spectrophotometer and a Hall Effect measurement system. Not only sufficient crystal structure but also excellent optical and electrical properties were obtained by this rapid laser annealing process for CIGS thin film. Acknowledgement: This work (Grants No. C0267866) was supported by Business for Academic-industrial Cooperative establishments funded Korea Small and Medium Business Administration in 2015.

**FP-20** Production of Ag Clusters by Plasma Gas Condensation and their Incorporation in an a:C Sputtered Matrix, *I Carvalho*, University of Coimbra, Portugal; *Sandra Carvalho*, University of Minho, Portugal; *A Cavaleiro*, University of Coimbra, Portugal

Ureteral stents are essential tools in contemporary urologic practice. However, urethral stents present complications at the level of microbial colonization leading to recurrent infections, being of extreme importance the development of new materials. In this way, the main propose of this study is to develop a new material aiming to improve biocompatibility, corrosion resistance, elasticity and, most especially, to reduce or eliminate adverse reactions in the body due to microbial colonization. A silver delivery device composed of a DLC coating embedded with Ag clusters, covering the existing stent material, is proposed.

Firstly, it is necessary evaluate the Ag clusters formation with respect to their size and shape since these parameters will controls the silver ion release and consequently the antimicrobial performance. In this sense, Ag clusters were produced by plasma gas condensation process consisting in a gas aggregation cluster source connected to a main deposition chamber. The work pressure in the main chamber and gun was 4 x10<sup>-3</sup> Pa and 100 Pa, respectively to produce Ag clusters, which will be incorporated in a:C matrixes with a current density applied of 2.4 up to 9.2 mA cm<sup>-2</sup>.

The clusters growth was evaluated in static and rotation mode. Current density applied to Ag target was kept constant at 7.6 mA cm<sup>-2</sup>. TEM results show that the deposition in rotation mode (2 rpm) leads to a decrease in the clusters mean size, 25 nm to the static mode to 15 nm to the rotate mode. In both modes, the clusters shape is spherical, however in the rotate mode the clusters appear to aggregate. The crystalline structure of Ag clusters was evaluated by the selected area diffraction patterns and the results show the formation of fcc crystalline Ag.

**FP-22** Biocompatible Thin Film Intermetalic Ti<sub>3-x</sub>AuO<sub>x</sub>, Vladimir Vishnyakov, University of Huddersfield, UK; *B Beake*, Micro Materials Ltd, UK; *J Devitt*, University of Huddersfield, UK

Titanium-gold intermetallics (both materials being biocompatible) are well suited as a replacement for hard tissue, and represents an opportunity to develop new medical applications in the field of arthritis, rheumatic and musculoskeletal diseases. It has been reported by other groups that intermetallics have the additional advantage, in right composition and crystallinity, of having a hardness value as high as 8 GPa. This high degree of hardness makes material exceptionally valuable for load-bearing body implants, for example. It is also know that the incorporation of relatively small amounts of oxygen, at the level of few atomic percent, can lead to titanium hardening. On the other hand, a small percentage of oxygen is almost inevitably incorporated in the titanium thin films during Physical Vapour Deposition (PVD). Ion sputtering of elemental titanium and gold targets with argon has been used to deposit Ti<sub>3-x</sub>AuO<sub>x</sub> (where 0.03<x<0.1) onto various substrates in pure Ar or Ar/O atmosphere. Energy Dispersive X-Ray Spectroscopy (EDX) and X-Ray Diffraction (XRD) have been used to assess the film composition and crystalline structure. Even at the highest level of investigation, the oxygen content in the films remain crystalline and its hardness can increase to almost 10 GPa. In addition, nanoscratching reveals certain loss of ductility as oxygen content increases.

FP-24 Features of Incident Particle Flux determining Growth Rates and Electrical Properties of Indium Tin Oxide Films Deposited by Ion-plating with dc Arc Discharge, *Hisashi Kitami*, *T Sakemi*, *Y Aoki*, Sumitomo Heavy Industries, Ltd., Japan

We have investigated the factors limiting growth rates and electrical properties of indium tin oxide (ITO) films on glass substrates (@ 200 °C)

deposited by ion-plating with dc arc discharge. We clarified the incident particle fluxes during film growth under the deposition conditions; the pressure in the deposition chamber of 0.18 - 0.71 Pa and the discharge current of 150 A. The resulting thickness of the ITO films was 150 (±3 %) nm. The carrier concentration of the ITO films was  $1.0 \times 10^{21}$  (±3 %) cm<sup>-3</sup>.

We measured the incident particle fluxes of the neutral atoms and ions for each species at the substrate level using a mass-energy analyzer (Hiden, EQP300), a Langumuir probe and a diaphragm gauge during the deposition. To clarify the factors limiting the growth rate and Hall mobility ( $\mu$ -H) of ITO films, we analyzed the relationship between the growth rates,  $\mu$ -H, and the incident fluxes of In species and O species.

As the pressure increased, the fluxes of In<sup>+</sup> (>10 eV) ions and O<sup>+</sup> (> 10eV) ions decreased and the fluxes of In<sup>+</sup> (<10 eV) ions and O<sup>+</sup> (< 10eV) ions increased. We found that the fluxes of In<sup>+</sup> (>10 eV) ions were dominant factor to limit the growth rate. The sticking coefficient of In species should be dependent on the incident energy into the substrate. As the fluxes of In<sup>+</sup> (> 10eV) ions and/or O<sup>+</sup> (> 10eV) ions increased,  $\mu_{\text{-H}}$  increased. The  $\mu_{\text{-H}}$  was governed by the microstructure and carrier concentration of the films, which originated in the incident energy fluxes (> 10eV) of In<sup>+</sup> ions and/or O<sup>+</sup> ions under the current deposition conditions. We will discuss it in more detail.

FP-25 Development and Microstructure Characterization of Single and Duplex Nitriding of UNS S31803 Duplex Stainless Steel, *L Varela*, University of São Paulo, Brazil; *C Pinedo*, Heat Tech & University of Mogi das Cruzes, Brazil; *H Dong*, *X Li*, University of Birmingham, UK; André Tschiptschin, University of São Paulo, Brazil

The microstructural changes involved in single Low Temperature Plasma Nitriding - (LTPN) and Duplex Nitriding (DN): High Temperature Gas Nitriding (HTGN) + Low Temperature Plasma Nitriding (LTPN), surface treatments of UNS S31803 were studied. Specimens of UNS S31803 duplex stainless steel (DSS) were (i) single low temperature plasma nitrided (LTPN) and (ii) duplex nitrided (DN) using high temperature gas nitriding (HTGN), followed by low temperature plasma nitriding (LTPN). In both cases, LTPN was carried out at 400°C for 20 h, in a 75%N<sub>2</sub>+25%H<sub>2</sub> atmosphere. HTGN was carried out at 1200°C, under a 0.1 MPa high purity N<sub>2</sub> gas atmosphere, during 8 hours. The microstructure of the as received material was composed by ferrite and austenitic stringers, aligned in the rolling direction. The results showed that LTPN of the UNS S31803 duplex stainless steel promotes the formation of a duplex modulated structure composed by 2.5  $\mu$ m thick, 1509 HV hard, expanded ferrite ( $\alpha_N$ ) regions, and 3.0  $\mu$ m thick, 1362 HV hard, expanded austenite  $(\gamma_N)$  regions on ferrite and austenite grains, respectively. Intense coherent ε-Fe<sub>3</sub>N nitride precipitation inside expanded ferrite was observed. ε-Fe<sub>3</sub>N nitrides precipitated with an orientation relationship [111]  $\alpha_N$  // [120]  $\epsilon\text{-Fe}_3N$ , leading to increased microhardness of the expanded ferrite regions. After the first step of the duplex nitriding treatment (HTGN) a 550  $\mu$ m thick, 330 HV hard, nitrogen rich, fully austenitic layer formed at the surface of the specimens, by transformation of ferrite stringers into austenite. The second nitriding step (LTPN) led to the formation of a homogeneous expanded austenite layer, 1144 HV on top of the thick fully austenitic layer, formed during the first step. The duplex treatment resulted in a more homogeneous, precipitatefree, microstructure and a better transition between the mechanical properties of the hardened outermost layer and the softer substrate.

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