



Study of wear, hardness and nanoindentation of magnetron-sputtered Inconel 625 coated titanium grade 5

Kunle Babaremu^{a*}, Tien-Chen Jen^a, Oluseyi Oladijo^{a,b}, and Esther Akinlabi^c

^aMechanical Engineering Department, University of Johannesburg, Auckland, South Africa

^bDepartment of Chemical, Materials and Metallurgical Engineering, Botswana International University of Science and Technology, Palapye, Botswana

^cDepartment of Mechanical and Construction Engineering, Faculty of Environment, Northumbria University, Newcastle, United Kingdom

Corresponding author: babaremunle10@gmail.com

Abstract

The properties of materials are often enhanced for applications in areas automotive, marine, aerospace, petrochemical and oil industrials where components are regularly exposed to atmospheric contaminant and corrosives, which eventually leads to loss in mechanical properties such as wear resistance, hardness and nanoindentation resistance. In view of these challenges, titanium grade 5 was coated with Inconel 625 using the magnetron sputtered coating technique, varying the process parameters such as temperature, power and deposition time. The chamber utilized for the magnetron sputtering was appropriately vacuumed at a base pressure of 1.13×10^{-5} mbar. The wear characteristics of the Inconel coated titanium samples using tribometer of 8.1.8 version in accord with ASTM G99 criterion, at a normal load of 10 N. A nanoindenter was employed to study the micro-hardness of the samples at 400.0 Hz in accordance with ASTM A-370. The indentation work done during plastic deformation ($W_{\text{plast.}}$) and elastic deformation ($W_{\text{elast.}}$) was also examined. Relative to the other test samples, the ICT C sample (sample coated at the temperature of 200°C, time of 90 minutes and power of 200 W) possessed the least wear rate of 0.001064 mm³/N/m. Similarly, the ICT C sample exhibited the highest micro hardness, as indicated by the Vickers hardness value which is 544.57 $\mu\text{N}/\text{mm}^2$. More so, the ICT C sample exhibited the lowest $W_{\text{elast.}}$ and $W_{\text{plast.}}$ values. These results indicated that the ICT C sample exhibited the lowest material loss, superior plasticity and elasticity, higher strength and great resistance to nanoindentation.

Keywords: Inconel 625; Micro-hardness; Nanoindentation; Sputtering and Titanium

1. Introduction

Modification of the surface of engineering components against deterioration is crucial in automotive, marine, aerospace, petrochemical and oil industrials [1, 2]. Surface modifications protect metals from wear, corrosion and structural failure, which could shorten their life span [3, 4], especially on exposure to contaminants and contact with counter bodies. In recent times surface modification methods such as arc welding and thermal spraying have been widely employed on metals to enhance their surface properties [5, 6]. However, magnetron sputtering deposition provides metals minimal distortion, strong bonding of coating with substrate and low dilution of substrate [7]. In magnetron sputtering coating technique, there is rarely crack formation, unlike the case of cladding processes where the propensity to crack formation is high as a result of rapid melt pool solidification [8, 9], which could be detrimental to the microstructure and corrosion performances of metals due to the ability of the cracks to act as regions for pitting and crevice corrosion [10, 11]. Although, further researches have revealed that with the optimization of the process parameters, addition of high ductility alloys and preheating of the parent metal before deposition, surface cracking in laser clad can be minimized [12, 13].

Inconel 625 possesses outstanding ductility and excellent corrosion resistance [14, 15]. The aforementioned properties of Inconel 625 make it a reliable and choice material for the modification of metal surfaces for oil and gas, automotive and marine industries [16, 17]. Thus, components and tools life span are enhanced in deteriorating environments by the sputtering of Inconel 625 on the surface of metals before being subjected to engineering applications [18, 19]. Inconel 625 has often found applications in where exceptional corrosion resistance and high temperature strength are required [20, 21]. Inconel 625 is a nickel-chromium based superalloy, whose nickel and chromium content offers high resistance in an oxidising environment. Also, the chromium content has the ability to passivate the surface of metals

continuously to form chromium oxide, which is the basic reason for its high corrosion resistance [22-24]. It was also reported that the exceptional ductility of Inconel 625 aids its propensity to solidification cracking prior to welding, especially when coated on metals through the sputter coating technique [25, 26].

Relative to most techniques of coating, sputter coating technique has the ability to give cleaner production environment, enhanced surface quality, superior efficiency of deposition and low wastage of materials, which leads to the improvement of the production economy [27, 28]. The formation of pores due to air bubbles entrapment is significantly low in the sputtered coating method. This has been severally reported with the Inconel sputtered coatings [29, 30]. More so, besides the remarkable microstructure of a typical Inconel 625 coated metals, they also often possess high corrosion resistance and outstanding micro-hardness in corrosive environments [31]. As a result of the above-mentioned characteristics of Inconel alloy and the magnetron sputtering deposition technique, Inconel 625 was coated on titanium grade 5 in this present work, varying the process parameters. The wear resistance, Vickers hardness and nanoindentation of the uncoated and coated samples were eventually investigated.

2. Experimental Procedure

Inconel thin coatings were developed on titanium substrate using a magnetron sputtering. The chamber utilized for the magnetron sputtering was appropriately vacuumed at a base pressure of 1.13×10^{-5} mbar. The deposition was accomplished at a time ranging from 60 to 90 minutes. The Sputtering of the samples was carried out at the power rating ranging from 100W to 200W. After completing the procedure of target thin film on the titanium substrate, the entire samples were kept in chamber to cool sufficiently and thereafter, the samples were removed from the vacuum and cautiously machined to a dimension of 10 mm x 10 mm for characterization. The sputtering process parameters are indicated in Table 1.

Table 1: Sputtering process parameters

Sample	Temperature		
	°C	Time (min)	Power (W)
Ti			
(Control)	-	-	-
ICT A	100	90	100
ICT B	150	90	150
ICT C	200	90	200
ICT D	150	60	150

2.1 Characterization of Sample

The wear characteristics of the Inconel coated titanium samples using tribometer of 8.1.8 version in accord with ASTM G99 criterion. The tribometer is a reciprocating sliding wear testing machine. It uses a 6 mm diameter ball made of hard alloyed steel as counter material. The wear characteristics of the samples were examined with a normal load of 10 N at the target temperature, lab temperature, frequency, humidity and linear speed of 24 °C, 21.48 °C, 15 Hz, 47.78%, and 0.25 cm/s, respectively. With the aid of the tribometer, the wear rate ($\text{mm}^3/\text{N}/\text{m}$) were examined. A nanoindenter was employed to study the micro-hardness of the samples at 400.0 Hz in accordance with ASTM A-370. The diamond hard tip was pressed on the test samples at a peak load of 300 mN for loading and 900 mN/min for the unloading. The Vickers hardness (HV) of the samples was also studied using the nanoindenter. Also, from the nanoindentation study, the values of force (mN) were plotted against the displacement (nm) or the indentation depth. The force-indentation depth plots indicated the indentation work done during plastic deformation (W_{plast}) and elastic deformation (W_{elast}), and the total work done (W_{total}) in picojoule (pJ). 1E-12 J is equivalent to 1pJ.

3. Results and Discussion

3.1 Wear rate of the uncoated and Inconel coated samples

Figure 1 indicated the wear rate of the uncoated (control) and Inconel coated samples. Compared to the entire test samples, the uncoated sample (titanium grade 5) exhibited the highest wear rate of $0.005795 \text{ mm}^3/\text{N}/\text{m}$. The wear rate was observed to reduce on the addition of Inconel 625. The wear rate of the ICT D sample (sample coated at the temperature of 150°C , time of 60 minutes and power of 150 W) was marginally better than that of the uncoated titanium. The wear rate of the ICT D sample was $0.004564 \text{ mm}^3/\text{N}/\text{m}$.

However, highly improved wear rates were observed with the ICT A sample (sample coated at the temperature of 100°C , time of 90 minutes and power of 100 W) and ICT B sample (sample coated at the temperature of 150°C , time of 90 minutes and power of 150 W). Relative to the other test samples, the ICT C sample (sample coated at the temperature of 200°C , time of 90 minutes and power of 200 W) possessed the least wear rate of $0.001064 \text{ mm}^3/\text{N}/\text{m}$. This indicated that the ICT C sample exhibited the lowest material loss [32, 33]. The inclusion of Inconel and the choice of process parameters could also have influenced the drastic reduction in wear rate of ICT C sample. The low wear rate of Inconel coated titanium samples compared to the uncoated sample indicated that the coated samples exhibited minimal volume loss per unit distance [34]. The Inconel coating could also have enhanced the lubricity of the surface of the titanium, hence, reducing slide friction which eventually resulted in the reduction of wear rate [35, 36].

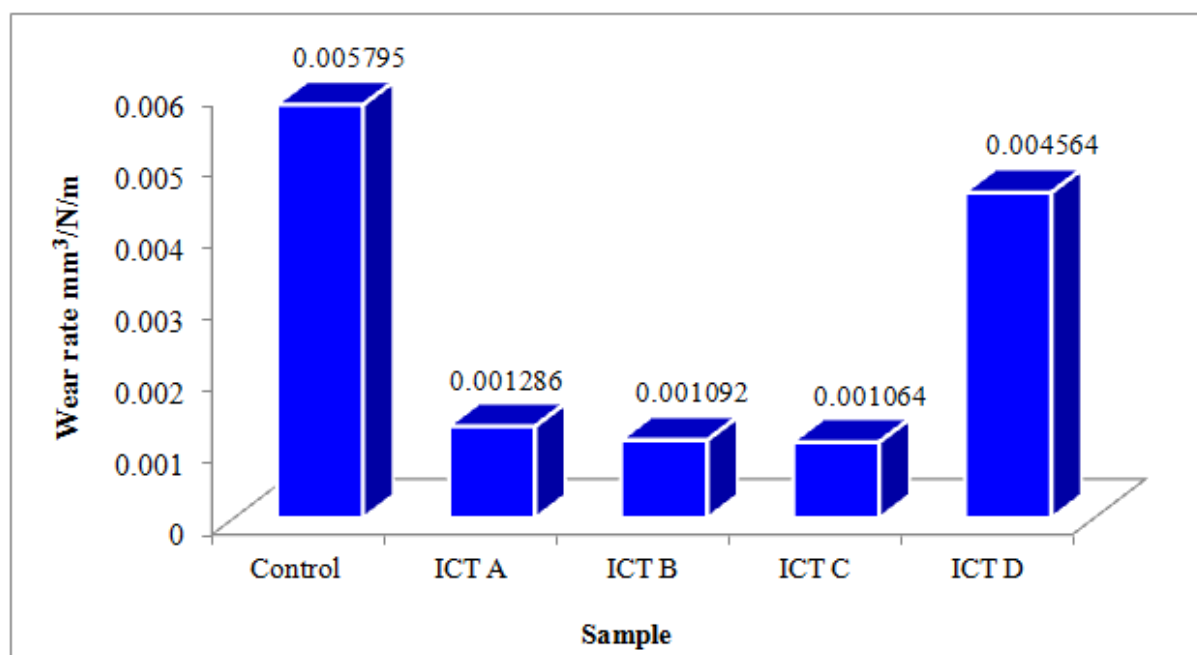


Figure 1: Wear rate of the Uncoated and Inconel coated samples

3.2 Micro hardness of the uncoated and Inconel coated samples

The micro hardness of the uncoated and Inconel coated samples is indicated in Figure 2. Compared to the other test samples, the ICT C sample (sample coated at the temperature of 200°C, time of 90 minutes and power of 200 W) exhibited the highest micro hardness, as indicated by the Vickers hardness value which is 544.57 μ N/mm². This showed that the ICT C sample provided maximum resistance to impression or indentation when a load was applied [37, 38]. The other Inconel coated titanium sample also exhibited higher Vickers hardness compared to the uncoated titanium sample. This indicated that the uncoated titanium sample offered the least resistance to the impressing force of the indenter. The relatively high micro hardness of the ICT C sample indicated the possibility of high plasticity, elasticity and strenght of the sample [39-41].

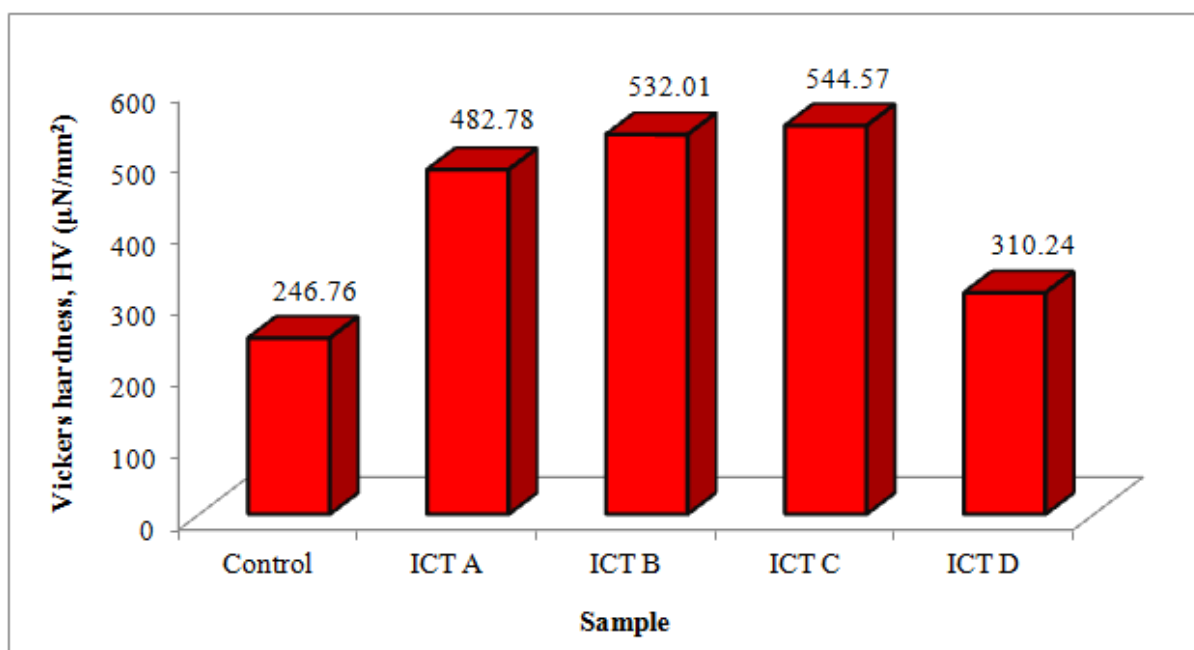


Figure 2: Micro hardness of the uncoated and Inconel coated samples

3.3 Work done analysis from the nanoindentation of the test samples

The work done for plastic and elastic deformation to occur in the uncoated and Inconel coated titanium samples are indicated in Table 2 and Figure 3. The work done during elastic deformation ($W_{\text{elast.}}$) and work done during plastic deformation ($W_{\text{plast.}}$) are indicated by the enclosed section bcd and bdea, respectively for the ICT D sample. The corresponding sections can also be derived on the other graphs. From Table 2 it can be seen that the uncoated titanium (control) exhibited the highest $W_{\text{elast.}}$ and $W_{\text{plast.}}$ of 350.55 pJ and 1174.33 pJ, respectively, which culminated to a higher W_{total} of 1524.88 pJ. This indicated that on the application of force, more indentation depth with minimal resistance was achieved on the uncoated titanium compared to the Inconel coated titanium [43, 44]. Figure 3 also confirmed the large $W_{\text{elast.}}$ and $W_{\text{plast.}}$ section of the uncoated titanium sample (control). However, relative to the entire the ICT C sample (sample coated at the temperature of 200°C, time of 90 minutes and power of 200 W) exhibited the lowest $W_{\text{elast.}}$ and $W_{\text{plast.}}$ value as shown Table 2. The $W_{\text{elast.}}$ and $W_{\text{plast.}}$ section were also confirmed to be very small for the ICT C sample. This indicated that lower indentation distance or depth was achieved by

the indenting material on the ICT C sample[45-47]. Generally, the reduced $W_{elast.}$ and $W_{plast.}$ values and sections of the Inconel coated titanium sample indicated that the sample provided higher resistance to nanoindentation, which led to the reduction in elastic and plastic deformation of the samples on the application of forces.

Table 2: Work done analysis from the nanoindentation result of the uncoated and Inconel coated titanium samples

Sample	$W_{elast.}$ (pJ)	$W_{plast.}$ (pJ)	W_{total} (pJ)
Control	350.55	1174.33	1524.88
ICT A	259.94	237.95	497.89
ICT B	254.27	168.85	423.12
ICT C	284.05	78.950	363.00
ICT D	300.61	890.38	1190.99

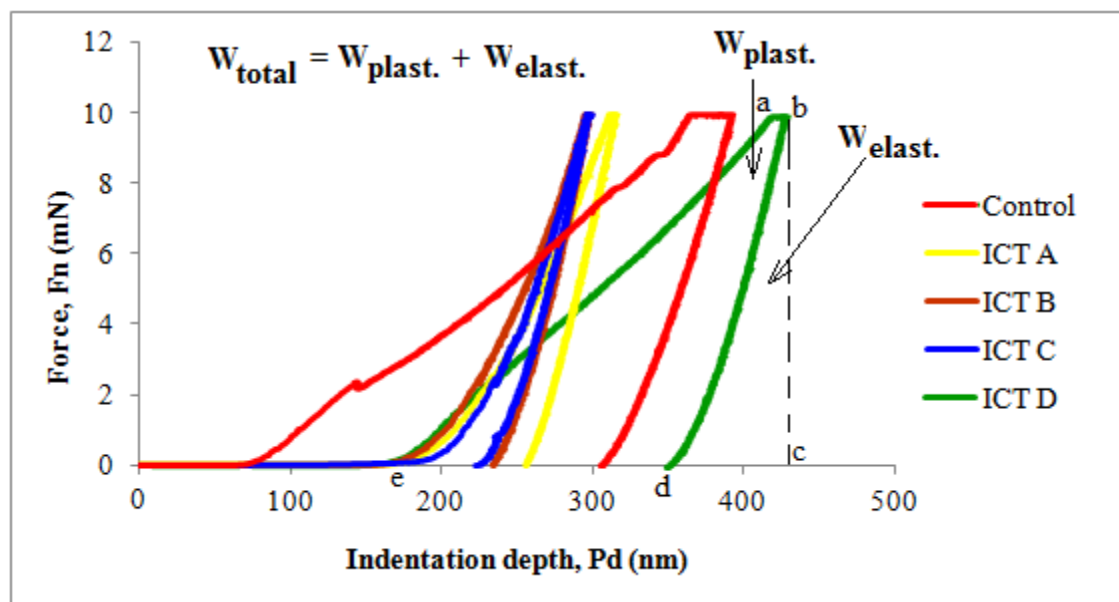


Figure 3: Plots of Force versus indentation depth for the uncoated and Inconel coated titanium samples

4. Conclusions

The hardness, wear and nanoindentation of the Inconel 625 coated titanium grade 5 accomplished through magnetron sputtered deposition route was studied. The Inconel coated titanium were observed to exhibit superior hardness, wear and nanoindentation resistance compared to the uncoated titanium. Specifically, the ICT C sample (sample coated at the temperature of 200°C, time of 90 minutes and power of 200 W) possessed the least wear rate of 0.001064 mm³/N/m. Similarly, the ICT C sample exhibited the highest micro hardness, as indicated by the Vickers hardness value which is 544.57 μN/mm². More so, the ICT C sample exhibited the lowest $W_{elast.}$ and $W_{plast.}$ values. These results indicated that the ICT C sample exhibited the lowest material loss, superior plasticity and elasticity, higher strength and great resistance to nanoindentation. Therefore, the Inconel coated titanium can be applied for several advanced applications in automotive, marine, aerospace, petrochemical and oil industrials.

Acknowledgement

The authors appreciate the University of Johannesburg for financial support through the University Research Committee (URC).

References

- [1] Chourasia, S., Tyagi, A., Pandey, S. M., & Murtaza, Q. (2023). A Critical Review of Thermal-Barrier Coatings and Critical Examination on Post Heat Treatment. *Recent Trends in Mechanical Engineering: Select Proceedings of PRIME 2021*, 845-852.
- [2] Hanagadakar, M. S., & Kulkarni, R. M. (2023). Environmental impacts and benefits of ceramic coatings. In *Advanced Ceramic Coatings*, 461-487.
- [3] Kumar, S., & Kumar, R. (2022). Recent Advances in Design and Fabrication of Wear Resistant Materials and Coatings: Surface Modification Techniques. *Handbook of Research on Tribology in Coatings and Surface Treatment*, 87-117.

- [4] Yuan, W., Xia, D., Wu, S., Zheng, Y., Guan, Z., & Rau, J. V. (2022). A review on current research status of the surface modification of Zn-based biodegradable metals. *Bioactive materials*, 7, 192-216.
- [5] Aramide, B., Pityana, S., Sadiku, R., Jamiru, T., & Popoola, P. (2021). Improving the durability of tillage tools through surface modification—a review. *The International Journal of Advanced Manufacturing Technology*, 116(1-2), 83-98.
- [6] Łatka, L., & Biskup, P. (2020). Development in PTA surface modifications—a review. *Advances in Materials Science*, 20(2), 39-53.
- [7] Alishahi, M., Mahboubi, F., Khoie, S. M., Aparicio, M., López-Elvira, E., Méndez, J., & Gago, R. (2016). Structural properties and corrosion resistance of tantalum nitride coatings produced by reactive DC magnetron sputtering. *RSC advances*, 6(92), 89061-89072.
- [8] Zhou, S., Huang, Y., Zeng, X., Hu, Q. (2008). Microstructure characteristics of Ni based WC composite coatings by laser induction hybrid rapid cladding. *Mater. Sci. Eng.: A* 480 (1–2), 564–572.
- [9] Zhou, S., Zeng, X., Hu, Q., Huang, Y. (2008). Analysis of crack behavior for Ni-based WC composite coatings by laser cladding and crack-free realization. *Appl. Surf. Sci.* 255 (5, Part 1), 1646–1653.
- [10] Abioye, T. E., McCartney, D. G., & Clare, A. T. (2015). Laser cladding of Inconel 625 wire for corrosion protection. *Journal of Materials Processing Technology*, 217, 232-240.
- [11] Zhang, P., & Liu, Z. (2016). Physical-mechanical and electrochemical corrosion behaviors of additively manufactured Cr-Ni-based stainless steel formed by laser cladding. *Materials & Design*, 100, 254-262.
- [12] Thawari, N., Gullipalli, C., Katiyar, J. K., & Gupta, T. V. K. (2021). Influence of buffer layer on surface and tribomechanical properties of laser cladded Stellite 6. *Materials Science and Engineering: B*, 263, 114799.

- [13] Tamanna, N., Crouch, R., & Naher, S. (2019). Progress in numerical simulation of the laser cladding process. *Optics and Lasers in Engineering*, 122, 151-163.
- [14] Shankar, V., Rao, K. B. S., & Mannan, S. L. (2001). Microstructure and mechanical properties of Inconel 625 superalloy. *Journal of nuclear materials*, 288(2-3), 222-232.
- [15] Guo, L., Zheng, H., Liu, S., Li, Y., Feng, C., & Xu, X. (2016). Effect of heat treatment temperatures on microstructure and corrosion properties of Inconel 625 weld overlay deposited by PTIG. *Int. J. Electrochem. Sci*, 11(7), 5507-5519.
- [16] Madhankumar, S., K. Manonmani, V. Karthickeyan, and N. Balaji. "Optimization of ultimate tensile strength of welded Inconel 625 and duplex 2205." *Journal of Mechanical Engineering and Sciences* 15, no. 1 (2021): 7715-7728.
- [17] Ceritbinmez, F., Günen, A., Gürol, U., & Çam, G. (2023). A comparative study on drillability of Inconel 625 alloy fabricated by wire arc additive manufacturing. *Journal of Manufacturing Processes*, 89, 150-169.
- [18] Ren, Y., Zhou, G. S., & Li, D. G. (2018). A pre-passive state observed for the passive film formed on Alloy 625 in a hydrochloric acid solution. *Applied Surface Science*, 431, 197-201.
- [19] Akande, I. G., Oluwole, O. O., Fayomi, O. S. I., & Odunlami, O. A. (2021). Overview of mechanical, microstructural, oxidation properties and high-temperature applications of superalloys. *Materials Today: Proceedings*, 43, 2222-2231.
- [20] Chail, G., & Kangas, P. (2016). Super and hyper duplex stainless steels: structures, properties and applications. *Procedia Structural Integrity*, 2, 1755-1762.
- [21] Gonzalez, J. A., Mireles, J., Stafford, S. W., Perez, M. A., Terrazas, C. A., & Wicker, R. B. (2019). Characterization of Inconel 625 fabricated using powder-bed-based additive manufacturing technologies. *Journal of Materials Processing Technology*, 264, 200-210.

- [22] Günen, A., Gürol, U., Koçak, M., & Çam, G. (2023). Investigation into the influence of boronizing on the wear behavior of additively manufactured Inconel 625 alloy at elevated temperature. *Progress in Additive Manufacturing*, 1-21.
- [23] Antonialli, A. Í. S., Carvalho, M. R. D. D., & Diniz, A. E. (2020). On the machinability of the Ni-30 high-temperature iron-based superalloy. *Journal of the Brazilian Society of Mechanical Sciences and Engineering*, 42, 1-11.
- [24] Zhang, S., Bian, T., Mou, L., Yan, X., Zhang, J., Zhang, Y., & Liu, B. (2023). Alloy design employing Ni and Mo low alloying for 3Cr steel with enhanced corrosion resistance in CO₂ environments. *Journal of Materials Research and Technology*, 24, 1304-1321.
- [25] David, S. A., Siefert, J. A., DuPont, J. N., & Shingledecker, J. P. (2015). Weldability and weld performance of candidate nickel base superalloys for advanced ultrasupercritical fossil power plants part I: fundamentals. *Science and Technology of Welding and Joining*, 20(7), 532-552.
- [26] Mohan Kumar, S., Rajesh Kannan, A., Pravin Kumar, N., Pramod, R., Siva Shanmugam, N., Vishnu, A. S., & Channabasavanna, S. G. (2021). Microstructural features and mechanical integrity of wire arc additive manufactured SS321/Inconel 625 functionally gradient material. *Journal of Materials Engineering and Performance*, 30, 5692-5703.
- [27] Ahmad, M. S., Pandey, A. K., & Abd Rahim, N. (2017). Advancements in the development of TiO₂ photoanodes and its fabrication methods for dye sensitized solar cell (DSSC) applications. A review. *Renewable and Sustainable Energy Reviews*, 77, 89-108.
- [28] Das, A., Das, S. R., Panda, A., & Patel, S. K. (2022). Experimental investigation into machinability of hardened AISI D6 steel using newly developed AlTiSiN coated carbide tools under sustainable finish dry hard turning. *Proceedings of the Institution of Mechanical Engineers, Part E: Journal of Process Mechanical Engineering*, 236(5), 1889-1905.

- [29] Iyyakkunnel, S., Marot, L., Eren, B., Steiner, R., Moser, L., Mathys, D., ... & Meyer, E. (2014). Morphological changes of tungsten surfaces by low-flux helium plasma treatment and helium incorporation via magnetron sputtering. *ACS applied materials & interfaces*, 6(14), 11609-11616.
- [30] Saqib, M., Beshchasna, N., Pelaccia, R., Roshchupkin, A., Yanko, I., Husak, Y., ... & Orazi, L. (2022). Tailoring surface properties, biocompatibility and corrosion behavior of stainless steel by laser induced periodic surface treatment towards developing biomimetic stents. *Surfaces and Interfaces*, 34, 102365.
- [31] Lont, A., Poloczek, T., & Górka, J. (2022). The structure and properties of laser-cladded Inconel 625/TiC composite coatings. *Materials Science-Poland*, 40(4), 91-103.
- [32] Harish, U., Mruthunjaya, M., Yogesha, K., Siddappa, P., & Anil, K. K. (2022). Enhancing the performance of inconel 601 alloy by erosion resistant WC-CR-CO coated material. *J. Eng. Sci. Technol*, 17, 0379-0390.
- [33] Kamboj, N., & Thakur, L. (2022). A study of processing and high-temperature sliding wear behaviour of Inconel-625 alloy TIG weld cladding. *International Journal of Materials Engineering Innovation*, 13(3), 191-207.
- [34] Günen, A. (2020). Properties and high temperature dry sliding wear behavior of boronized Inconel 718. *Metallurgical and Materials Transactions A*, 51, 927-939.
- [35] Montazeri, S., Aramesh, M., & Veldhuis, S. C. (2020). Novel application of ultra-soft and lubricious materials for cutting tool protection and enhancement of machining induced surface integrity of Inconel 718. *Journal of Manufacturing Processes*, 57, 431-443.
- [36] Musfirah, A. H., Ghani, J. A., & Haron, C. C. (2017). Tool wear and surface integrity of inconel 718 in dry and cryogenic coolant at high cutting speed. *Wear*, 376, 125-133.

- [37] Verdi, D., Garrido, M. A., Múñez, C. J., & Poza, P. (2015). Cr₃C₂ incorporation into an Inconel 625 laser cladded coating: effects on matrix microstructure, mechanical properties and local scratch resistance. *Materials & Design*, 67, 20-27.
- [38] Verdi, D., Garrido, M. A., Múñez, C. J., & Poza, P. (2014). Mechanical properties of Inconel 625 laser cladded coatings: Depth sensing indentation analysis. *Materials Science and Engineering: A*, 598, 15-21.
- [39] Luo, X. T., Yao, M. L., Ma, N., Takahashi, M., & Li, C. J. (2018). Deposition behavior, microstructure and mechanical properties of an in-situ micro-forging assisted cold spray enabled additively manufactured Inconel 718 alloy. *Materials & Design*, 155, 384-395.
- [40] Singh, R., Schrufer, S., Wilson, S., Gibmeier, J., & Vassen, R. (2018). Influence of coating thickness on residual stress and adhesion-strength of cold-sprayed Inconel 718 coatings. *Surface and coatings technology*, 350, 64-73.
- [41] Singh, R., Schrufer, S., Wilson, S., Gibmeier, J., & Vassen, R. (2018). Influence of coating thickness on residual stress and adhesion-strength of cold-sprayed Inconel 718 coatings. *Surface and coatings technology*, 350, 64-73.
- [42] Zhao, L., Alam, M., Zhang, J., Janisch, R., & Hartmaier, A. (2020). Amorphization-governed elasto-plastic deformation under nanoindentation in cubic (3C) silicon carbide. *Ceramics International*, 46(8), 12470-12479.
- [43] Dada, M., Popoola, P., Mathe, N., Adeosun, S., & Pityana, S. (2021). Investigating the elastic modulus and hardness properties of a high entropy alloy coating using nanoindentation. *International Journal of Lightweight Materials and Manufacture*, 4(3), 339-345.
- [44] Zak, S., Trost, C. O. W., Kreiml, P., & Cordill, M. J. (2022). Accurate measurement of thin film mechanical properties using nanoindentation. *Journal of Materials Research*, 37(7), 1373-1389.

- [45] Zak, S., Trost, C. O. W., Kreiml, P., & Cordill, M. J. (2022). Accurate measurement of thin film mechanical properties using nanoindentation. *Journal of Materials Research*, 37(7), 1373-1389.
- [46] Movassagh-Alanagh, F., & Abdollah-Zadeh, A. (2023). Effects of B/C ratio on the structural and mechanical properties of TiBCN coating deposited by PACVD. *Ceramics International*, 49(16), 26191-26204.
- [47] Rominiyi, A. L., & Mashinini, P. M. (2023). Nanoindentation study of mechanical and wear properties of spark plasma sintered Ti-6Ni-xTiCN composites. *Ceramics International*, 49(2), 2194-2203.