Incorporation of Impact Erosion and Corrosion Behavior of Multi-layer Metallic Nitride Structures Deposited on Various Metal Substrates into a Lab-Based Class

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Abstract

Erosion caused by the impingement of solid particles that are carried in a transport fluid on to a metal surface continues to be a common problem. The hardness of the metal, the angle of particle impact, and the intensity of the impact determine the magnitude of the erosional process. A group of international students from the Brazil Scientific Mobility Program (BSMP) were placed on a team that allowed them to investigate impact erosion on various metals using a custom designed erosion apparatus built by students. These metals include various steel and aluminum alloys that have been enhanced through heat treatment and/or multi-layer structures consisting of alternating ceramic (metallic nitride) and metallic coatings deposited on the metal substrates using a DC high vacuum magnetron sputtering deposition machine. This experimental study allowed students to learn about metal enhancement techniques, use current material evaluation equipment, and then compare the changes to the material properties on the surface of the specimen tested with respect to the erosion process. What is presented is a summary of the student's learning experience with respect to the science of plasma deposition, its performance during the erosion process, and how this work can be used in a junior/senior mechanical engineering laboratory class.

Keywords

Laboratory experience, erosion, testing, fluid flow, multi-layered structure

Introduction

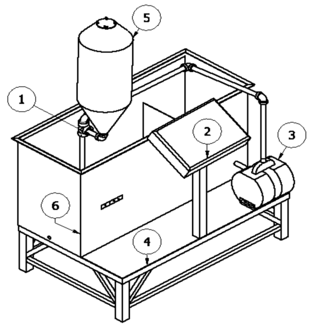
Erosion of metal components when exposed to sand laden slurries continue to be a problem in many industries[1]. The cost associated with replacement of eroded parts and the downtime needed when replacing these components often result in large losses to corporations. The ability to model erosional effects on metal components accurately can aid in the reduction of the cost associated with the losses by allowing these metal components to be placed in optimal locations and to allow for replacement to occur before catastrophic failure occurs. Two groups of students performed research in this area. The first group was a set of traditional Mercer undergraduate/graduate students who looked at the initial phases of the research. The second group of students were from the Brazil Scientific Mobility Program (BSMP) were brought together from different universities around the states to examine the effect of erosion on different metals. The BSMP is a one-year, non-degree program for Brazilian students to study in the United States. BSMP is part of the Brazilian government's larger initiative to grant Brazilian university students the opportunity to study abroad at US colleges and universities, by offering scholarships to students in the Science, Technology, Engineering and Mathematics (STEM) fields. After completion of an academic year including a summer internship, students return to Brazil to complete their degrees. Four students accepted the program to come and perform research at Mercer University. All were at different universities and no experience in regards to the project and testing that they were about to undertake. The purpose of this project was to expose the students to a research experience in a 10-week session and to consider how to implement his into a lab based class at Mercer.

Background

The process of materials degradation during functioning is a continuous problem for equipment working in harsh environmental conditions. Their mechanical performance at the surface can be approached by hardness, wear resistance, erosion and corrosion measurement and analysis. For example, the study of materials response under laboratory investigated erosion is a good simulation of materials behavior offering the possibility of studying and optimizing their response to aggressive environments. Erosion evaluation is an important and complex phenomenon that is observing the corrosive action of a liquid (e.g. water) simultaneously with the wear abrasion of the small and hard particles (e.g. sand) that are suspended in the flowing liquid; therefore erosion testing has both a wear and an impact testing component. Depending on the angle of attack, velocity, the impinging liquid and suspended solid particles are causing severe wear damages on the surface, similar to the damages caused by operation in a dusty environment (e.g. for car panels, aircraft blades, pipelines) [1-5] that will be initiation sites for future corrosion development through pits. Two common industrial used materials were investigated under erosion laboratory simulated conditions: 4140 alloy steel and 6061 aluminum alloy. The abrasive wear and impact caused by liquid and sand striking the surface at different angles and pressures, using the room temperature were evaluated. In order to observe improvement of the surface materials response, the 4140 alloy steel was heat treated (classical corresponding heat treatment) and tested in similar conditions. During the recent years, multilayer hard ceramic coatings are extensively used in industry for wear and corrosion protection and are potential candidates for erosion protection[6-16]. However, the impact component that is acting along with the wear during the erosion testing is posing a supplementary problem due to the high brittleness of the hard ceramic coatings. There is a change in the "brittle" to "ductile" response of the material depending on the material removal process, i.e. changes in response with impact angle. Beside erosion, there is also a complex interaction of the material surface with the corrosive liquid and the multilayer hard ceramic coating are offering a good corrosion protection and accordingly a better erosion response from materials surface. The research investigated the erosion behavior of 4140 alloy steel and 6061 aluminum alloy coated with a bilayer combination of titanium and titanium nitride ceramic nanolayers.

Test Apparatus

The testing apparatus for the students is the erosion test fixture that was designed by a Senior design group at Mercer University. At Mercer University, seniors complete a two semester senior design sequence where they must first determine a project and perform the analysis of building the project and secondly executing the manufacturing and testing of the apparatus. The design group constructed the erosion test fixture and a schematic of the test fixture shown in Figure 1.



Item Number	Part Description
1	Auger
2	Control Box
3	Diaphragm Pump
4	Frame
5	Sand Hopper
6	Settling Tank

Figure 1: Erosion Test Fixture Schematic

Figure 2: Erosion Test Fixture

Plasma Vapor Deposited Coatings

The samples were coated with a high vacuum magnetron sputtering device shown in Figure 4. This device uses argon gas and an electric field to create a plasma discharge inside of the chamber. Argon ions are attracted to the cathode as shown in Figure 6 to dislodge atoms of the target material. Material ejected from the target is then coated on objects within the vacuum chamber.

Figure 3: High Vacuum Magnetron Sputtering Device

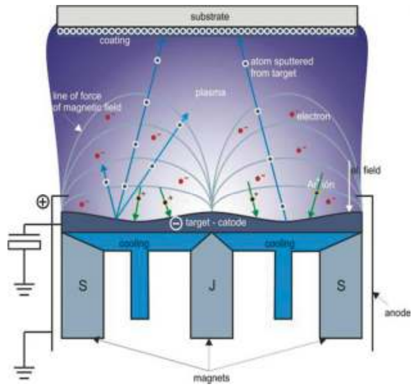


Figure 4: Sputtering Process[17]

Metal samples are placed in the chamber and are coated with the metal atoms displaced from the target. In this study, various metals were coated using either titanium or titanium nitride which was created with in a nitrogen rich environment. The thickness of the coating was verified with a profliometer.

Experimental Procedure

Students were tasked with testing various metals: tool steel H13, 4140 steel, stainless steel and aluminum. For each material there was at least one coated sample and one or more non-coated sample tested. Before coating the samples, the hardness of the material was checked. The aluminum samples had a hardness value of 60 HRB, the tool steel H13 had a hardness value of 90 HRB, and the 4140 and stainless steels were harder registering 28 and 50 on the HRC scale. The 4140 steel and H13 were heat treated and tempered to even harder values to replicate what will be done in the lab class.

It is anticipated that in the lab based class, the students would perform the following set of experiments which could be varied based on the group. The parameters listed show the permutations on the set of experiments that could be run.

- Angle of Impact: 45^o from the vertical axis of the nozzle, could vary from 90^o to 30^o
- Metal Type: HT13, 4140, Al 6061, and Stainless Steel
- Coating Type: TiN Coating with varying thickness layers, 4000 Å and 11000 Å tested
- Sand Volume: 20, 40, 50, 100, 200, 300, and 400 ml
- Air Pressure: Set at 30 psi
- Nozzle Size: 0.157-in ceramic used for sand blasting applications

Measurements obtained for each sample

- Hardness of Sample using hardness tester
- Weight before testing using calibrated scale
- Weight before coating using calibrated scale
- Weight after testing using calibrated scale
- Maximum depth of cavity created using profilometer

- Area of cavity created using an optical microscope and/or profilometer

Student Learning

Both sets of students were able to master the art of the testing pretty quickly which bodes well for implementation in a lab-based class. Students were tasked with all process of the tester including pre and post experimentation exercises, data collection and data analysis. After a couple of tests with faculty present, students were able to quickly adapt. Depending on the amount of sand loaded in the apparatus, the test duration varied from a couple of minutes to 20 minutes with about 1 hour of preparation before testing and 1 hour of cleaning after the testing is done. While running many experiments in a lab based class would be difficult, this would work in either of the junior or senior lab sequence as one of the projects that are assigned. The students could run one or two samples with the apparatus and then analyze pretested samples with the equipment in the lab to understand the process and procedures outlined. Expected outcomes would be to have the students proficient in using modern lab equipment, data collection, and data analysis.

Experimental Data

Data collected during these sets of experiments are still in the early stages as the investigators learn the abilities of the test equipment and techniques. It is expected that the coatings would reduce the amount of erosion on the metals, and preliminary data does show a slight decrease in mass loss.

Table 1: Erosion Test Results

		Weight Before Coating (g)	Weight Before Testing (g)	Weight After Testing (g)	Weight Difference After Testing (Δ)	Hardness
4140	B1		54.6197	54.6063	0.0134	57 HRC
			54.6063	54.5959	0.0104	
			54.5959	54.5906	0.0053	
	C2		56.2111	56.1976	0.0135	60 HRC
			56.1976	56.1827	0.0149	
			56.1827	56.1701	0.0126	
	A	54.3336	54.3336	54.3308	0.0028	Light/Yellow TiN Coating; Coating Layer Thickness: 4000 Å
	N	54.9591	54.9591	54.9570	0.0021	
	B3	52.6090	52.6090	52.6022	0.0068	

Also the depth of the cavity created is less with the coating where the coated steel average cavity is on the order of 46 micrometers where the non-coated samples vary from 55 to 65 micrometers. All other relevant parameters show general trends.

Conclusions

The work shown here illustrates the ability to adapt a research topic into a lab based class, by exposing the students to experimental techniques that will be useful to them as they head into the workforce. This research allows the students to get hands-on experience with water/sand impact erosion and then allows them access to modern measurement techniques to learn how to assess the magnitude of the erosion. A future plan of this work is to build a wear testing apparatus that will allow the coatings to be evaluated in wear instead of just erosion. It is expected that the coatings will perform even better in wear. A senior design project has been launched and it is expected to be completed by the fall of 2017.

References

1 H.M. Slot, E.R.M. Gelinck, C. Rentrop, E. van der Heide, “Leading edge erosion of coated wind turbine blades: Review of coating life models”, Renewable Energy 80 (2015) 837-848.

2. Deng Jianxin, Wu Fengfang, Lian Yunsong, Xing Youqiang, Li Shipeng, “Erosion wear of CrN, TiN, CrAlN, and TiAlN PVD nitride coatings”, Int. Journal of Refractory Metals and Hard Materials 35 (2012) 10–16.

3 Y.P. Purandare, M.M. Stack, P.Eh. Hovsepian, “Velocity effects on erosion–corrosion of CrN/NbN “superlattice” PVD coatings, Surface & Coatings Technology 201 (2006) 361–370.

4. M.M. Stack, Y. Purandare, P. Hovsepian, “Impact angle effects on the erosion–corrosion of superlattice CrN/NbN PVD coatings”, Surface & Coatings Technology 188–189 (2004) 556– 565.

5. Shizhong Zhang, KimDam-Johansen, Pablo L Bernad Jr., SørenKiil, “Rain erosion of wind turbine blade coatings using discrete water jets: Effects of water cushioning, substrate geometry, impact distance, and coating properties”, Wear 328-329(2015)140–148.

6. E. Bemporad, M. Sebastiani, C. Pecchio, S. De Rossi, “High thickness Ti/TiN multilayer thin coatings for wear resistant applications”, Surface & Coatings Technology 201 (2006) 2155–2165.

7. L.A. Dobrzanski, K. Lukaszkowicz, “Erosion resistance and tribological properties of coatings deposited by reactive magnetron sputtering method onto the brass substrate”, Journal of Materials Processing Technology 157–158 (2004) 317–323.

8. Q. Yang, D.Y. Seo, L.R. Zhao, X.T. Zeng, “Erosion resistance performance of magnetron sputtering deposited TiAlN coatings”, Surface & Coatings Technology 188–189 (2004) 168–173.

9. Duhua Wang, YuhuA Ni, Qun Huo, Dennis E. Tallman, “Self-assembled monolayer and multilayer thin films on aluminum 2024-T3 substrates and their corrosion resistance study”, Thin Solid Films 471 (2005) 177– 185.

10. M.A. Ezazi, M.M.Quazi, E.Zalnezhad, AhmedA.D.Sarhana, “Enhancing the tribo-mechanical properties of aerospace AL7075-T6 by magnetron-sputteredTi/TiN,Cr/CrN&TiCr/TiCrNthin film ceramic coatings”, Ceramics International40(2014)15603–15615.

11. Ayyappan Susila Praveen, J. Sarangan, S. Suresh, J. Siva Subramanian, “Erosion wear behaviour of plasma sprayed NiCrSiB/Al2O3 composite coating”, Int. Journal of Refractory Metals and Hard Materials 52 (2015) 209–218.

12. Jianliang Lin, Jaeho Jang, In-wook Park, Ronghua Wei, “Structure and properties of CrSiCN coatings deposited by pulsed dc magnetron sputtering for wear and erosion protection”, Surface & Coatings Technology 287 (2016) 44–54.

13. Guojun Zhang, Bin Li, Bailing Jiang, Fuxue Yan, Dichun Chen, “Microstructure and tribological properties of TiN, TiC and Ti(C, N) thin films prepared by closed-field unbalanced magnetron sputtering ion plating”, Applied Surface Science 255 (2009) 8788–8793.

14. Y. Purandare, M.M. Stack, P. Hovsepian, “A study of the erosion–corrosion of PVD CrN/NbN superlattice coatings in aqueous slurries”, Wear 259 (2005) 256–262.

15. S. Hassani, J.E. Klemberg-Sapieha, M. Bielawski, W. Beres, L. Martinu, M. Balazinski, “Design of hard coating architecture for the optimization of erosion resistance”, Wear 265 (2008) 879–887

16. Krzysztof Szymański, Adam Hernas, Grzegorz Moskal, Hanna Myalska, “Thermally sprayed coatings resistant to erosion and corrosion for power plant boilers - A review”, Surface & Coatings Technology 268 (2015) 153–164.

17. E. Bergmann and D. Rosello, “Corrosion protection with perfect atomic layers”, POLYSURFACES #06 (2012).

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