

EVALUATION OF COATING PERFORMANCE BY NANO INDENTATION TECHNIQUE AND ITS VALIDATION BY FINITE ELEMENT APPROACH

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Abstract - To improve the surface properties of a substrate coating plays an important role. The performance of the coating layer over the substrate can be known under different load conditions. Finite element simulation is one of the techniques to validate the result where the improvement of the coating is done by optimization. Through FEM, very complex stress strain field of thin films or bulk material can be studied in nano indentation process. Aim of the present study is to model and simulate nano indentation process on DLC coated HSS material by finite element method and to compare its result with experimental results. The 3D axi symmetric model was modeled/meshed in static analysis using ANSYS software. The spherical ball indenter of diamond material with 200 μ m diameter was considered as rigid body. The model (substrate) has ability to deform the material permanently carried with plastic deformation during indentation under different loading-unloading condition. However, the unloading portion of the curve for most material consists mainly of elastic recovery. After the unloading process the unloading curve shows the final permanent deformation (plastic deformation). So, FEM could be the effective tool to find out the simulation of hardness and its verification. Developing load-displacement curve of FEM model which can be compared with load-displacement curve measured by the experiments. Total deformation is calculated under load-displacement graph from where the hardness of the material can be calculated mathematically. The FE simulation result shows that they are in better comparison with the experimental results. We are arriving at a conclusion by incrementing the velocity from 1mN/sec to reach to upto 5N max force to obtain the behavior of the material. The boundary condition is applied by fixing the specimen at horizontal axis. It is considered and assumed that the Contact between the ball indenter and the specimen are frictionless and there is only a normal pressure transmitted between the indenter and the specimen. Materials are also considered to be fully homogeneous and free from contaminates, pinholes and

defects and the Coating/substrate interface is perfectly bonded.

Key Words: Nano-indentation, ANSYS software, Surface coatings, Three-dimensional process.

1. INTRODUCTION

The mechanical properties of bulk materials have limitation with respect to wear resistance, hardness, scratch resistance, elastic modulus etc. Such properties can be improved by depositing appropriate coating on bulk materials. Coating is a controlled process at the nano level to significantly enhance the ability surface properties of substrate and improve the tribological performance of the products.

In engineering application, the ability of coating to perform without catastrophic failure is major concern. The failure of coated substrate can be related to the failure of coating itself or deformation of the substrate. Several different mechanisms of failure have been observed in coated surface, such as elastic, plastic and fracture deformation. It has been observed that coated surface very often fail due to tensile fracture. Tensile fracture mainly occurs at the contact edge of a contact due to the high tensile stresses experienced there. So, it is important to analyse the properties of coated surfaces.

Nano-indentation is one of the techniques which is used to measure mechanical properties of bulk materials and coated surface which are mentioned above. Different numerical techniques have been developed to use in many field of engineering that can be used in nano-indentation problems. One of the simple methods is indentation technique which has advantage over conventional techniques of easy use and excellent accuracy using minimum amount of materials. This technique is useful for welded parts with continuous property variation, brittle materials with unstable crack growing. But the test results are difficult to analyse because of the complicated triaxial stress state under the indenter.

However such a drawback can be eliminated by finite element analysis. Material deformations and the influence

of coating thickness and elastic modulus can be analysed by three-dimensional finite element method (FEM) modelling on microlevel, by stress, strain, and displacement computer simulations. Thus, finite element method provides numerical solution to the complex problems.

2. LITERATURE REVIEW

Lichinchi et al. (1998) studied simulation of Berkovich nano-indentation experiment on thin films, in which FE technique is applied for nano-indentation of thin coated substrate. This experiment was simulated in ABAQUS FE software package. For variable indentation depths numerical values hardness of TiN/HSS were calculated. [1]

Panich et al. (2004) studied nano-indentation of bulk materials FE method and compared with experimental results. A two-dimensional axi-symmetric model and three-dimensional model were developed using the ABAQUS software programme. The simulation results of load displacement curve shows close relation between experimental and FE result. [2]

Li and Beres (2006) studied a 3D finite element modelling for TiN coated titanium alloy substrate under scratch test. Rockwell C-scale indenter scratching a TiN/Ti-6Al-4V coated substrate system is considered with coulomb friction between the work surface and indenter. A Von Mises yield criterion was employed to determine plastic deformation. [3]

Holmberg et al. (2006a) found the modelling of stresses and strains by using ball indenter sliding on coated surface. A three-dimensional FE model is developed. Fracture and stress condition of coated substrate were found out by experimentation and compared with FE simulation results. The steel substrate material is elastic-plastic, the coating is linear elastic and indenter is rigid one. [4]

Holmberg et al. (2006b) studied deformation of material and Young's modulus in tribological analysis ball indenter sliding on a hard coated surface. The diamond ball indenter moves with increasing the load for tribological contact analysis of TiN coated steel surface. A 3-D FE model is developed and simulate for stress, strain and displacement. These values are then compared with experimental results. [5]

Heinrich et al. (2009) studied bulk materials for nano-indentation and multiple indenter tips for finding out the mechanical properties. It involves study of two different nano-indenters tips. The results show superior results as compared to one indenter. Friction between materials and indenter is included in the cases studied. [6]

Chicot et al. (2011) experimental and theoretical data from literature was used to study the mechanical properties of goethite, hematite and magnetite. Using indentation and

molecular dynamic analysis hardness and elastic properties of materials were obtained. [7]

Ziskind et al. (2011) showed nano-indentation test on peritubular and intertubular human dentin to find out Young's modulus. Two step experiments were carried out firstly on the intertubular dentin areas & highly mineralized peritubular dentin sections and secondly test nano-indentation experiment was done on the face of polyhedron specimen. [8]

Marteau et al. (2011) proposed a new method first contact detection errors on indentation size and hardness effect measurements. By prediction of the Bernhardt law and according to the gap between their shapes several nano-indentation loading curves simultaneous treatment was done. [9]

Ajaja et al. (2011) studied the Ti coatings which were deposited on Ti surface. Nano-hardness test and micro hardness test were performed on coated surface and bulk surface. For each sample true hardness values and nano-indentation measurements, plasticity of strain gradient were used. [10]

Hyun et al. (2011) studied a FE simulation of dual conical ball indenter method. The analysis of indentation parameter and load-depth curves was done. The diameter of projected contact is explained as a function of material properties; tip-radius, indenter angle and modulus of elasticity were found. [11]

Passeri et al. (2011) used the atomic force microscopy in order to study polyaniline thin film and indentation modulus. The atomic microscopy instrument is used to calculate indentation experiment at micro level. In order to get maximum indentation depth, polymeric reference materials, hardness values were calibrated. [12]

Ananth and Ramesh (2012) studied behaviour of non-conformal surfaces of ball on flat surface subjected to normal and the substrate shear stress. The substrate material chosen for the analysis is ultra high strength steel, titanium alloy, magnesium alloy, aluminium alloy and the stainless steel material are considered for the rigid ball material. Before imposing sliding the FE model is validated through the Hertz solution for different contacting material pair under normal load. The analysis facilitated in evaluating the surface phenomena variables. The results are obtained for the combining effect of normal load and shear friction and same were normalized with the corresponding yield strength to determine the failure mode of the material surface. The results obtained from the analysis are exploited in the understanding of the tribological behaviour of the contact pairs. [13]

Kaimin and Yue (2012) studied nano-indentation test on the influence of acid and alkaline on the spinal surface and nickel aluminates spinal. For evaluation of Young's modulus and hardness the products were tested in order to incorporating the waste materials. [14]

Giannakopoulos and Zisis (2013) showed finite element study on the adhesion less contact of flat surface by Knoop and Vickers indenter .The pressure sensitivity is modelled according the classic, perfectly,plastic potential of Drucker-prager. The micro indentation test were analysed and numerical results shows phenomenology of test. Young’s modulus, Knoop and Vickers hardness is measured and analysed. [15]

Pardo et al. (2013) studied properties of hard nano-composite coatings and self-organized a-C: Cu the effect of concentration of metal on mechanical tribological properties, mechanical and structural. In this experiment, 20-25nm thick copper metal layers and alternating carbon are used for developing multi layer self-organised structure. The nano-hardness and young’s modulus is found out. [16]

Chen et al. (2013) studied scratch damage resistance of silica-based on polymeric substrates coated withsol-gel coatings. By using the ISO 15184 standard scratch test were done. [17]

Luo et al. (2014) studied the elastic–plastic analysis of ultrafine-grained Si2N2O–Si3N4 composites by nano-indentation and finite element simulation. A theoretical method was used for calculating the stress–strain relationship of brittle ceramic materials based on the experimental nano-indentation test result. The finite element results were then compared the calculation and simulation results. [18]

Trueba et al. (2014) performed the fracture characterization of thin-films by dual tip indentation and presents a new fracture characterization technique for thin films, called dual tip indentation (DTI). [19]

Huang et al. (2014) studied the nano-indentation creep study on an ion beam irradiated oxide dispersion strengthened alloy and found out that Oxide dispersion strengthened (ODS) alloy is considered advanced materials for nuclear application due to their radiation tolerance and creep resistance. Ion beam irradiation is used to study the property changes due to displacement damage.

Tillmann and Momeni (2014) studied the deposition of layered composite thin films made of NiTi and TiCNfilms deposited Si (100) substrate by DC magnetron sputtering. The mechanical properties, microstructures, and shape memory behaviour of these bilayers were investigated using nano-indentation technique, differential scanning calorimetry, scanning electron microscopy andX-ray diffraction. The results of this research show that the presence of TiCN layer on NiTi thin film modifies its mechanical properties while maintaining the shape memory effects. [20]

Xiao et al. (2014) studied the four-ball test to find out tribological performance of WC/C, DLCand TiN coatings using and evaluated the tribological performance

of coatings for high-speed and heavy-duty power-transmitting gears. [21]

3. METHODOLOGY

3.1. Design of Experiment

Design of Experiment methods are used in robust case Design for obtaining product and process conditions. Taguchi methods are statistical methods developed by Genichi Taguchi to improve the quality of manufactured goods, and more recently also applied to engineering design Of Experiment

3.2 Plan of Experiment

In the present study L9 standard orthogonal array has been used which is attributed to its suitability for 3 level problems. On the basis of Taguchi method four factors with three levels of each are selected and, the L9 array has been made for calculating the parameters of PECVD Machine

Table -1: PECVD process parameters and their levels

Process Parameter	Units	Level
Voltage	Volts(V)	-50 -100 -150
Deposition Pressure	μbar	0.25 6 100
Frequency	KHz	2 4 6
Gas Composition	%	60:40 80:20 90:10

3.3 Experimental Setup

DLC films were deposited by using a standard inductively coupled plasma-enhanced chemical vapor deposition (IC-PECVD) reaction chamber powered by a 13.56 MHz RF power (50 W) source. A high speed steel of 10 mm x10 mm square area of 1.2 mm thickness was taken as substrate for depositing DLC film. The carbon source gas was methane (CH4), diluent was hydrogen. The pressure inside the chamber was varied by closing and opening the throttle valve. The total flow rate was maintained a constant at 10 sccm, by varying the composition of the precursor gases. The load-displaced curves were obtained by Nano-hardness Tester (CSEM Instruments) fitted with an integrated optical (Nikon)/Atomic Force Microscope (Surface Imaging Systems) equipped with a Berkovich diamond indenter, having a triangular-pyramid shape.

Table -2:L9 array table for DOE based on Taguchi Method.

Trial No.	Voltage	Freq.	Deposition Pressure	Gas Composition (%)
1.	-50	2	0.25	60:40
2.	-50	4	6	80:20
3.	-50	6	100	90:10
4.	-100	4	0.25	90:10
5.	-100	6	6	60:40
6.	-100	2	100	80:20
7.	-150	6	0.25	80:20
8.	-150	2	6	90:10
9.	-150	4	100	60:40

4. FE MODELLING

Table-3: Mechanical properties of Coating material, Indenter and Substrate

Sr. No	Name of the Modeling parts	Properties
1.	Ball indenter	Material=Diamond Young's modulus=1140Gpa Poisson's ratio=0.07

2.	Coating	<p>COATING 1 Material=DLC 1 Young's modulus=180Gpa Poisson's ratio=0.3</p> <p>COATING 2 Material=DLC 2 Young's modulus=195Gpa Poisson's ratio=0.3</p> <p>COATING 3 Material=DLC 3 Young's modulus=182Gpa Poisson's ratio=0.3</p> <p>COATING 4 Material=DLC 4 Young's modulus=178Gpa Poisson's ratio=0.3</p>
3.	Substrate	Material=HSS Young's modulus=200Gpa Poisson's ratio=.29

The values of applied load by the ball indenter on coated substrate are in the range of minimum of 0 N and maximum of 5 mN. The displacement values of all four coatings are as given in table no.--, which ranges according to the weight applied. The total deformation was confined within the coating film. From the Table it is clear that the residual displacement was 55 nm for a total displacement of 137 nm for indentation coating 1 and also the film went up till 60% elastic deformation and 40% plastic deformation. For coating of DLC 2 the residual displacement was 46 nm for a total displacement of 133 nm and that the film went up till 65% elastic deformation and 35% plastic deformation. The residual displacement was 61 nm for a total displacement of 125 nm for indentation of coating of DLC 3 and that the film went up till 52% elastic deformation and 48% plastic deformation, and for the coating of DLC 4 residual displacement was 53 nm for a total displacement of 136 nm and that the film

went up till 62% elastic deformation and 38% plastic deformation.

The models were designated as master and slave surfaces. In these models, ball indenter was considered as rigid of diamond material having tip radius of 200 μm . The indentation is very small as compared to size of the sample. The volume of substrate was taken in to consideration as $10 \times 10 \times 1.2$ mm (length, width, and thickness). Input material properties required for modelling is shown in table 1. Figure 1 shows the substrate and DLC coating after meshing. The substrate was meshed by Hexa (Brick) mesh and the ball indenter was meshed by shell mesh with spider rigid. A fine mesh was used around the contact area and near the tip of indenter. The developed model has number of nodes are 109500 and number of element are 36194.

The following are the assumptions made to simulate and model nano-indentation process:

- Materials is homogeneous and continuum.
- During modelling surface are considered smooth and residual stresses are neglected.
- Coating/substrate interference is perfectly bonded.

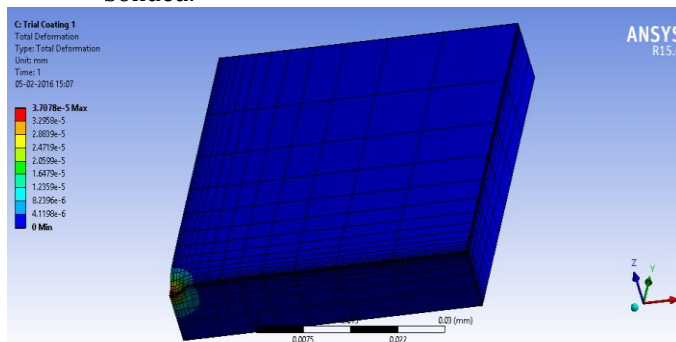


Fig -1: FEA results for coating 1

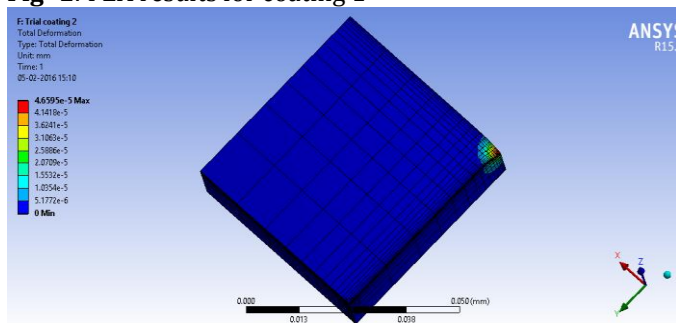


Fig -2: FEA results for coating 2

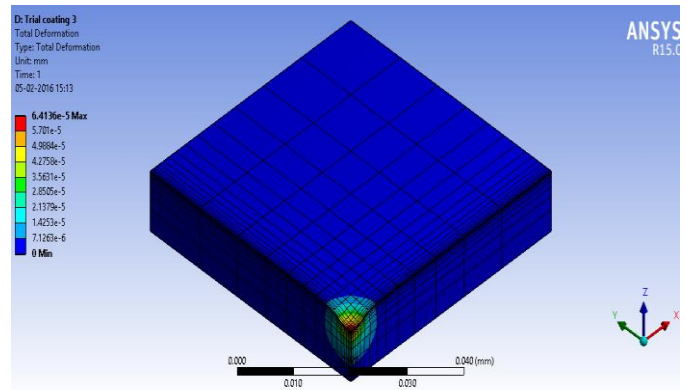


Fig -3: FEA results for coating 3

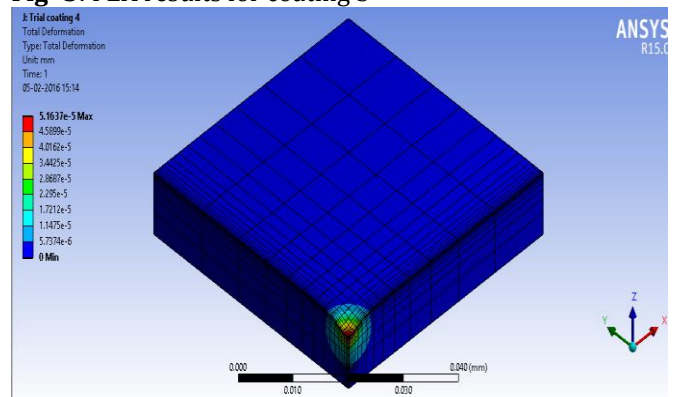


Fig -4: FEA results for coating 4

5. RESULTS

With the help of finite element analysis, the nanoindentation loading-unloading process of DLC coated HSS substrate was done. The comparison of load-displacement curves between experimental results and FE calculation is shown in chart 1,2,3,4. In load-displacement curves for all for coating trial the prediction from FE analysis are in good agreement with experimental results.

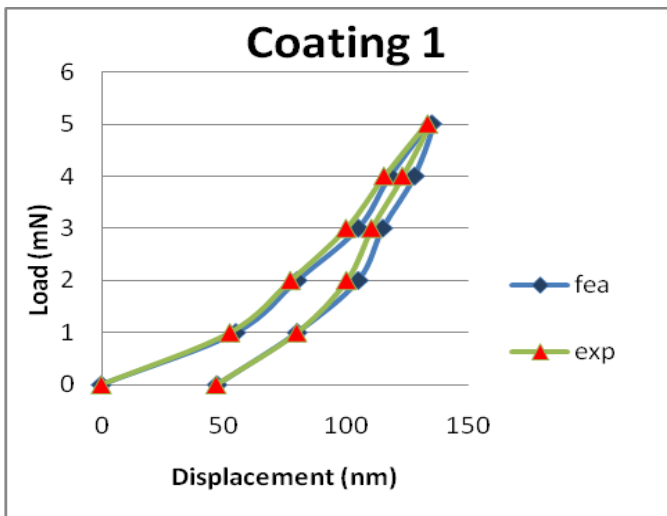


Chart -1: Graphical result of FEA and Experimental values of coating 1

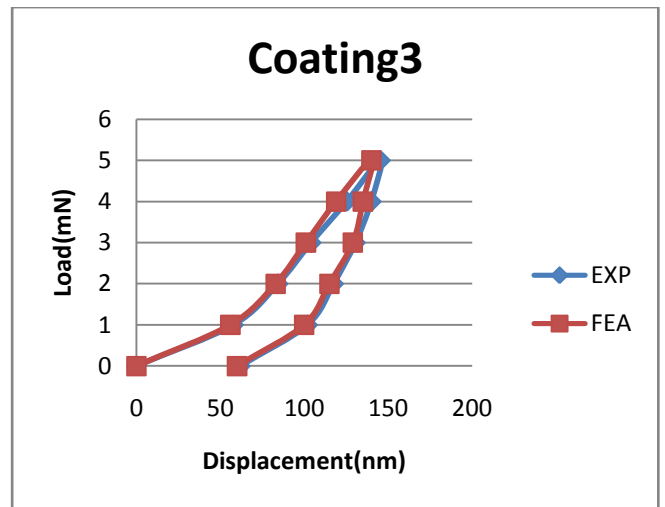


Chart -3: Graphical result of FEA and Experimental values of coating 3

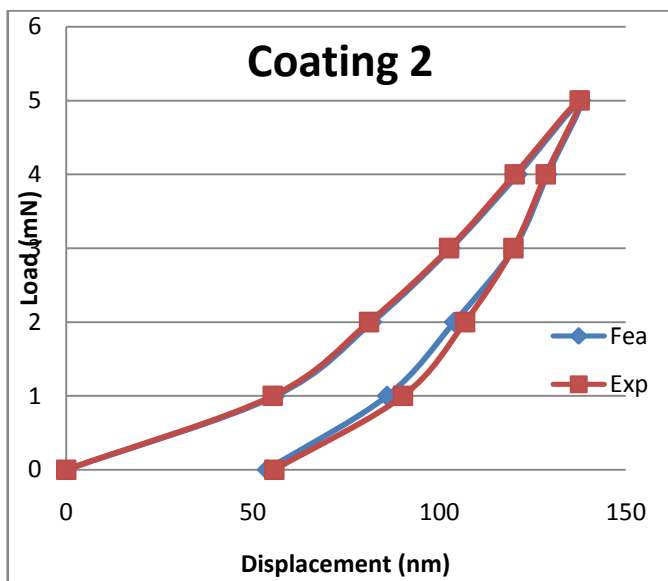


Chart -2: Graphical result of FEA and Experimental values of coating 2

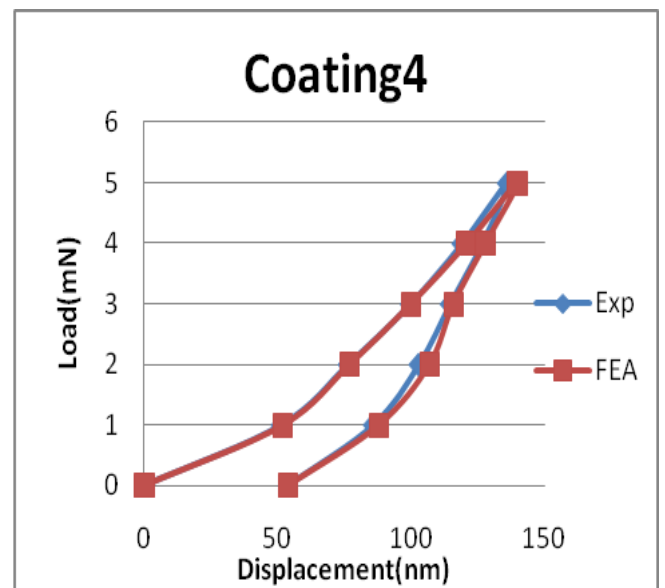


Chart -4: Graphical result of FEA and Experimental values of coating

Table -3: Comparison between FEA and Experimental Value for loading and unloading conditions

Load(mN)	Coating 1		Coating 2		Coating 3		Coating 4	
	FEA(nm)	EXP(nm)	FEA(nm)	EXP(nm)	FEA(nm)	EXP(nm)	FEA(nm)	EXP(nm)
0	0	0	0	0	0	0	0	0
1	55	52.514	56	55.307	56	57.8601	52	51.3301
2	80	77.2507	81.9902	81.1492	83	84.4064	77	76.3998
3	110	100.0172	103	102.6118	101	104.139	100	99.7463
4	130	115.5646	121	120.217	119	125.3639	121	119.1203
5	140	133.417	138	137.606	140	145.446	140	136.1489
4	130	123.0666	129	128.5397	135	139.9709	128	126.7145
3	100	110.43	120	119.8488	129	130.5591	116	114.7015
2	95	100.21	104	106.8913	115	117.5394	107	102.9793
1	90	79.870	86	90.2423	100	101.9802	88	85.8624
0	47.319	46.937	53.802	55.7566	60	61.8973	54	53.9324

6. CONCLUSION

In the present research work, finite element modeling and analysis of nano-indentation process is carried out. Elastic and plastic behavior of the material is studied using Finite element model for nano-indentation process. This model is tested for 4 trials Diamond Like Carbon(DLC) coating of high speed steel material. Finite element results were compared with experimental nano-indentation process. Based on the experimentation results and finite element simulation following conclusions were drawn:

1. Nano-indentation experiments were successfully carried out for all four Trial coating. Four runs were carried out on each samples at different position namely a, b, c, d which shows that all curves are in close deviation with each other.
2. During nano-indentation experiment it was observed that all nine Trial coating obtained 60% plastic deformation and 40% elastic deformation.
3. The FE simulation results are in good agreement with the experimental results as shown in table 3
4. The total Deformation (Permanent Deformation) obtained for each Trial Coating in FEA is in good comparison with the Experimental results.

REFERENCES

- [1] Chicot D., Mendoza J., Zaoui A., Louis G.; Mechanical properties of magnetite (Fe3O4), hematite (α-Fe2O3) and goethite (α-FeO.OH) by instrumented indentation and molecular dynamic analysis, Journal of the European Ceramic Society. 28, 2653-2573, 2011.
- [2] Ziskind D., Hasday M., Cohen S., Wagner H.; Young’s modulus of peritubular and intertubular human dentin by nanoindentation test, Structural Biology. 174, 23-30, 2011.
- [3] Ajaja J., Goldbaum D., and Chromik R.; Characterization of Ti cold spray coatings by indentation methods , ActaAstronautica, 69, 923-928,2011.
- [4] Hyun H., Kim M., Haeng J., Lee H.; A dual conical indentation technique based on FEA solutions for property evaluation, Mechanics of Materials, 43, 313-333, 2011.
- [5] Passeri D., Alippi A., Rossi M., Bettucci A.; Indentation modulus and polyaniline thin film by atomic force microscopy, Synthetic materials, 161, 7-12, 2011.
- [6] Ananth M., and Ramesh R.; Analysis of sliding behaviour of non-conformal surfaces subjected to shear traction, International Conference on Modelling Optimization and Computing. 38, 2645-2656, 2012.

- [7] Kaimin S. and Yue H.; Nano-indentation on nickel aluminate spinal and the influence of acid and alkaline on the spinal surface, *Ceramics International*. 38, 3121-3128, 2012.
- [8] Pardo A., Buijnsters J., and Endrino J.; Effect of the metal concentration on the structural, mechanical and tribological properties of self-organized a-C:Cu hard nano-composite coatings, *Applied Surface Sciences*, 280, 791-798, 2013.
- [9] Chen Z., Linda Y., and Wu A.; Scratch damage resistance of silica-based sol-gel coatings on polymeric substrates, *Tribology of Polymeric Nanocomposites*, 2, 467-511, 2013.
- [10] Zijing Huang, Nanoindentation creep study on an ion beam irradiated oxide dispersion strengthened alloy, *Journal of Nuclear Materials*, 451, 162-167, 2014.
- [11] Wolfgang Tillmann and Soroush Momeni, Deposition of superelastic composite NiTi based films, *Vacuum*, 104, 41-46, 2014.
- [12] Yangyi Xiao, Wankai Shi, Jing Luo, Yijian Liao., Tribological performance of TiN, WC/C and DLC coatings measured by the four-ball test, *Ceramics International*, 40(5), 6919-6925, 2014.