Optimization of process parameters in the RF-DC plasma N$_2$-H$_2$ for AISI420 molds and dies

Hengky Herdianto$^1$, D.J. Djoko H. Santjojo$^{2,*)}$ and Masruroh$^3$

Department of Physics, Faculty of Mathematics and Natural Science, University of Brawijaya, Veteran Street, Malang 65145, INDONESIA

E-mail: 1hengkyherdianto@gmail.com; 2dsantjojo@ub.ac.id; 3ruroh@ub.ac.id

*) Corresponding author

Abstract. The RF-DC plasma N$_2$-H$_2$ was used to make precise AISI420 molds and dies have complex textured geometry. The quality of the molds and dies directly affect the quality of the produced parts. The excellent examples of molds were used for injection molding lenses and dies used for the precision forging of automotive drive train components. In this study, a temperature, DC bias, and duration as process parameters of the RF-DC plasma N$_2$-H$_2$ have been optimized for molds and dies fabrication. The mask-less micro-patterned method was utilized to draw the initial 2D micro patterns directly onto the AISI420 substrate surface. The unprinted substrate surfaces were selectively nitrided by the RF-DC plasma N$_2$-H$_2$ at 673 K for 5400 s by 70 Pa with hollow cathode device. Energy Dispersive X-ray was utilized to describe the nitrogen content distribution at the vicinity of the border between the unprinted surfaces. This exclusive nitrogen mapping proves that only the unprinted parts of the substrate have high content nitrogen solutes. XRD analysis was performed to investigate whether the iron nitrides were precipitated by RF-DC plasma N$_2$-H$_2$ in the AISI420.

1. Introduction
The quality of molds and dies directly affect the quality of the produced parts. The excellent examples of molds were used for injection molding lenses and dies used for the precision forging of automotive drive train components [1]. The case-hardened AISI420, graded by STAVAX in the commercial is used as a mold for injection molding [2]. Micro- and nano-textures on the metal and polymer surfaces of parts and components have functions to reduce the friction and wear in [3,4] and to improve the joining strength in [5,6].

EDM (Electric Discharge Machining), laser machines [7], LIGA and photolithography process is the way to create micro and nano textures onto a substrate [8]. These four techniques have advantages in their applications but are expensive on an industrial. In the present study, the original 2D micro patterns were directly drawn onto the molds and dies surfaces. This printed molds and dies were subjected to the plasma N$_2$-H$_2$ by using the printed area as a mask. Since the masked area is free from the plasma N$_2$-H$_2$, the unmasked or unprinted area is selectively plasma nitrided and hardened.

After [2,7,9,10], the non-traditional processing was proposed to make micro-texturing onto molds and dies with aid of the RF-DC Plasma N$_2$-H$_2$. The process parameters of plasma N$_2$-H$_2$ are important factors for obtaining desired material properties. The species of plasma are a key point in the functionalization process of the metal surface for molds and dies micro pattern.
Properly selected parameters can improve species of plasma and material properties e.g. surface hardness, wear resistance, corrosion resistance [11,12], and suitability [13]. The process parameters of plasma N\textsubscript{2}-H\textsubscript{2} consist of temperature, gas flow rate, stress, pressure, and duration [14]. In those previous works, was found optimal parameters [7,9,10] that can be explained in Table 1.

Table 1. Summarizes the experimental process parameters for RF-DC plasma N\textsubscript{2}-H\textsubscript{2} in those previous works [7,9,10]

| Process        | Parameters                                                                 |
|----------------|-----------------------------------------------------------------------------|
| Pre-sputtering | DC (-500 V), RF (0 V), pressure (75 Pa), temperature (693 K), duration (1800 s), and carrier gas (N\textsubscript{2} only) |
| Nitriding      | DC (-300 V), RF (250 V), pressure (75 Pa), temperature (693 K), duration (14400 s), carrier gas (N\textsubscript{2} + H\textsubscript{2}), and with partial pressure (N\textsubscript{2} = 100 ml/min; H\textsubscript{2} = 20 ml/min) |

The used DC bias at -500 voltages and 1800 s in the pre-sputtering process have a negative impact on the quality of masks that have been printed using Ink-Jet printing technology with carbon ink. Masks undergo layer vanished so that it looks faded both at the edge and on the micro pattern. The temperature was used at 693 K could potentially increase the precipitation of CrN and Cr\textsubscript{2}N. According to [15] and [16], the precedence of CrN and Cr\textsubscript{2}N were hard and wear-resistant characteristics but reduces corrosion resistance. So the low-temperature nitriding process can be avoided and minimize chromium migration.

The used gas pressure 75 Pa and 14400 s also increased the rate of vanished on the mask layer. In this study, a temperature, DC bias, and duration as process parameters of the RF-DC plasma N\textsubscript{2}-H\textsubscript{2} have been optimized for molds and dies fabrication. The possibility of fine micro-texturing by the plasma N\textsubscript{2}-H\textsubscript{2} must be demonstrated by using the well-aligned micro-patterns.

2. Experiment

2.1. Maskless patterning with use of nano-carbon ink

The 2D micro patterns were drawn onto the AISI420 specimen and molds. Figure 1 depicts a typical micro-pattern drawn on the AISI420 sheet with use of the nano-carbon ink. Even when using the same CAD data for micro patterning, there were two manners in drawing: the positive-image drawn as depicted in Figure 1a and the negative-image drawn as shown in Figure 1b.

Figure 1. Micro patterned onto the AISI420 sheet surface: a) positive image, b) negative image
At present, the drawing area was limited by 25 x 25 mm with the spatial resolution down to 1 μm. The various kinds of micro-units with geometric complexity were straightforwardly designed by CAD and drawn on the AISI420 substrates. This mask-less micro-patterning was useful to draw any designed micro-head pattern with the complex geometries and within the high spatial resolution.

2.2. RF-DC plasma N\textsubscript{2}-H\textsubscript{2} system
The RF-DC plasma N\textsubscript{2}-H\textsubscript{2} was employed for selective nitrogen concentration into their unprinted part. RF-DC plasma N\textsubscript{2}-H\textsubscript{2} system was set-up for selective nitriding and hardening of AISI420. Different from the PECVD (Plasma Enhanced Chemical Vapor Deposition) or DC-pulse nitriding, where the plasmas were ignited and generated at the frequency of 13.56 MHz or its multiples, the present RF-DC plasma N\textsubscript{2}-H\textsubscript{2} system has no mechanical matching box with a slow response time of 1 s to 10 s to adjust the applied power. Since both the input and output powers were automatically matched by frequency adjustment around 2 MHz, the matched response time was only limited to 1 ms at most. This prompt power control provides to make full use of mesoscopic plasma pressure range over 50 Pa.

![Figure 2. RF-DC plasma N\textsubscript{2}-H\textsubscript{2} system: a) outlook of system, and, b) schematic illustration of system](image)

The differences from the conventional processes, the vacuum chamber was electrically neutral so that RF-power and DC-bias should be controlled independently from each other. A dipole electrode was utilized to generate RF-plasma; DC bias was directly applied to the specimens. Heating unit was located under this DC-biased cathode plate. In the following nitriding experiments, the specimens were located on the cathode table before evacuation down to the base pressure of 0.1 Pa. Then, nitrogen gas was first introduced as a carrier gas for heating. After heating to the specified holding temperature, the nitrogen pre-sputtering was started at the constant pressure. After pre-sputtering, the hydrogen gas was added to nitrogen gas with the specified partial pressure ratio. In particular, the partial pressure ratio of nitrogen to hydrogen gases is constant by 5 to 1 as a standard condition by controlling the gas flow rate to be 100 ml/min for N\textsubscript{2} gas and 20 ml/min for H\textsubscript{2} gas. Both of pressure (P) and temperature (T) controls were automatically performed with the tolerance of ΔP < 1 Pa in deviation of partial pressure and ΔT < 1 K in temperature fluctuation.

2.3. Observation and measurement
The plasma condition was observed and identified by using the optical emissive-light spectroscopy (EOS; PMA-11, Hamamatsu, Co. Ltd). SEM (Scanning electron microscope; Shimadzu, Corp) was used to observe printed mask pattern of the specimen before nitriding. EDX (Energy Dispersive X-ray; SSX-550, Shimadzu, Co. Ltd.) was utilized to analyze the nitrogen content distribution at the vicinity of the border between the printed and unprinted surface. The hardness of nitride specimen was measured by Vickers micro hardness testing (Mitsutoyo, Co. Ltd) while XRD (Smartlab, Rigaku, Co., Ltd.) was used for analyzing the microstructure of specimen.
3. Results and discussion

3.1. Species diagnostic of RF-DC plasma N$_2$H$_2$

Through the quantitative plasma diagnosis, the generated species in the plasmas were detected and monitored by EOS. Figure 3 and Table 2 shows an emissive light spectrum measured in the plasmas for the pre-sputtering process (DC with N$_2$ only) and nitriding process (RF-DC with N$_2$ and H$_2$). NH, as well as the activated nitrogen atom and molecules, were detected in both spectra. With the use of the mixed nitrogen and hydrogen gases, every peak of intensity was enhanced even with a little addition of hydrogen gas. The species of N$_2^+$, N$_2$, N$_2^*$, N-1, and NH might be responsible for high infiltration of nitrogen atoms into the AISI420.

![Figure 3. The EOS of plasmas N$_2$H$_2$](image)

| Species | Transition | $\lambda$ (nm) | Reference |
|---------|------------|----------------|-----------|
| N$_2^+$ | $B^2\Sigma_u^+ \rightarrow \chi^3\Pi_u^+$ (1→1) | 388.43 | [17] |
|        | $B^2\Sigma_u^+ \rightarrow \chi^3\Pi_u^+$ (0→0) | 391.4 | [18] |
|        | $B^2\Sigma_u^+ \rightarrow \chi^3\Pi_u^+$ (1→2) | 423.65 | [19] |
|        | $B^2\Sigma_u^+ \rightarrow \chi^3\Pi_u^+$ (0→1) | 427.81 | [19] |
| N$_2$  | C$^2\Pi_u \rightarrow B^2\Pi_u$ (0→0) | 337.1 | [18] |
|        | C$^2\Pi_u \rightarrow B^2\Pi_u$ (1→0) | 353.67 | [17] |
|        | C$^2\Pi_u \rightarrow B^2\Pi_u$ (0→1) | 357.69 | [19] |
| N-1    | 2s$2p^2$(P)$_3^1\rightarrow 2s2p^2$(D)$_5^1$ | 425.01 | [17] |
| NH     | $\lambda^2\Sigma \rightarrow \lambda^2\Sigma$ (0→0) | 336.0 | [18] |

3.2. Optimization of the DC bias and duration in the pre-sputtering process

The combination of RF and DC sources produce high state plasma because RF field creates extended plasmas and used together with the enhanced sputtering and biasing effect of the DC source. This effect is expected to increase the effectiveness of nitriding process and save energy consumption [22]. DC bias at -500 voltage and 1800 s in the pre-sputtering process [7,9,10] have a negative impact on the quality of masks that have been printed using Ink-Jet printer with carbon ink. In the present study, shows that pre-sputtering parameters with DC bias at -450 volts and duration at 400 s can minimize the carbon layer degradation. The content of carbon layer lasts in the range of 10-15 wt%.

3.3. Optimization of the temperature and duration in the nitriding process

The variations in the pre-sputtering and nitriding process temperatures were used 573 K, 593 K, 613 K, 633 K, 653 K, 673 K, and 693 K. The results were shown in Figure 4, same as previous research resulted [22] with increasing temperature, the deposited nitrogen atom at substrate surface has enough energy and longer chance to form homogeny of nitride layer, and therefore the maximum surface hardness increases as depicted in Figure 5. As nitriding temperature is increased the vibrations of atoms in the substrate will increase thereby increasing the diffusivity of nitrogen into the substrate so that the thickness of the nitride layer increases.
Figure 4. a) Distributions of nitrogen solute in the depth from the surface of the masked and unmasked region on the cross-section, b) micro-hardness depth profiles of RF-DC plasma $N_2-H_2$ onto AISI420 specimens for different temperature

Figure 4a shows concentration depth profiles of nitrogen elements mapping measured by Energy Dispersive X-ray (EDX) on samples the cross-section. The concentration of the nitrogen near the surface reached up to 11.54 wt% and decreased drastically at a depth of 60 μm. Micro-Vickers hardness testing was performed to describe the surface hardness distribution as well as the hardness depth profile. The applied load was constant by 0.50 N (or 50 g). Decreasing of nitrogen concentration has an impact on hardness reduction as depicted in Figure 4b.

Figure 5b depicts the measured micro-Vickers hardness profile along the scanning yellow line in Figure 5a. The measured hardness was nearly equal to the original matrix hardness of 348 Hv in the masked regions of the specimen surface. The average hardness reaches 1468 Hv in the unmasked or the nitrided regions. A sharp change in hardness from 348 Hv up to 1468 Hv was detected in Figure 5b across the border between the masked and unmasked regions.

Figure 5. a) AISI420 specimen after nitriding and polishing, b) measured hardness profile along the yellow scanning line in Figure 5a

The diffusion of nitrogen significantly increase and nitriding process more effective if temperature approaching 500°C. This result [22] was due to the fact that Cr precipitates and reacts with N to form CrN at temperatures above 400°C and it increases when the temperature approaches 500°C. At higher temperature Cr precipitates stronger so that nitrogen has longer chance to react with Cr to produce CrN and $Cr_2N$. Although at a temperature of 693 K is more hardness than 673 K, this optimization selects 673 K due to minimizing CrN and $Cr_2N$. The precipitation of CrN and $Cr_2N$ not only have a hard and wear-resistant characteristic but also low corrosion resistance [23].
The duration of RF-DC plasma N$_2$-H$_2$ is the most effective at 5400 s and condition of the micro pattern was maintained with good quality on this parameter. In the present study, shows that nitriding parameters with duration at 5400 s can minimize the carbon layer degradation. The content of carbon layer lasts in the range of 7.63 wt%. Figure 6b depicts the measured micro-hardness depth profiles the original hardness that near surface reached up to 1214 Hv and while it remained to be 335 Hv at a depth of 60 μm from the surface.

![Figure 6. a) Content of carbon layer for different nitriding duration, b) micro-hardness depth profiles of RF-DC plasma N$_2$-H$_2$ onto AISI420 specimens for different nitriding duration](image)

Figure 7 shows that N$_2^+$ species (388.43 nm) and N$_2^+$ (391.40 nm) have consistently increased when nitriding process temperature was increased. In addition, NH species tend to be stable at a high intensity if compared with N$_2$, N$_2^+$, and N-1 species. So the most plausible plasma species in the nitriding process along with temperature optimization are N$_2^+$ and NH.

![Figure 7. a) Effect of nitriding temperature on intensity of plasma species, b) effect of nitriding duration on intensity of plasma species](image)

The formation of N$_2^+$ species occurs by the ionization reaction chain N$_2$ + e$^-$ → N$_2^+$ + 2e$^-$. The N$_2^+$ species act directly on the Fe substrates surfaces at AISI420 through a diffusion process. The NH species are the result of the dissociation process by reaction N + H$_2$ → NH + H. In addition, NH is formed by ionization by reaction N$^+$ + H$_2$ → NH + H$. The role of NH as a damper from the oxide surface will increase the diffusion of nitrogen atoms. Additionally, saturates trapped nitrogen to increase the diffused species. Species formed between nitrogen and hydrogen neutralizes the barrier effect of surface oxidation during diffusion.
Geometry and dimensions were observed and measured by SEM after ink jet printing and RF-DC plasma \( \text{N}_2-\text{H}_2 \) in the present parameters that were optimized. Energy Dispersive X-ray analysis was utilized to describe the nitrogen content distribution at the vicinity of the border between the unprinted surface. As depicted in the first group of Figure 8a, 8b, 8c and second group of Figure 8g, 8h, 8i, the nitrogen maps selectively on the unprinted surface area both in the positive and negative images. No nitrogen was presented in the printed areas. On the other hand, the carbon from the ink maps only on the printed surface area in both images. This exclusive carbon-nitrogen mapping proves that only the unprinted parts of the substrate should have high content nitrogen solutes.

**Figure 8.** Printed micro positive model onto the AISI420 (a, b, c), printed micro negative model onto the AISI420 (g, h, i), nitrogen distribution of micro-patterns positive model onto the AISI420 (d, e, f), and nitrogen distribution of micro-patterns negative model onto the AISI420 (j, k, l)

The hardness profile was measured along a single yellow scanning horizontal line across the micro-pattern between the printed and unprinted regions. In order to demonstrate that the original printed pattern should be homogeneously transformed into the hardness profile pattern, a single square region was only left unprinted at the center of AISI420 specimen as shown in Figure 9a and 9b.

Figure 9c compares the hardness profiles measured both in the lateral and longitudinal directions across the mask. The less significant difference was seen in both hardness profiles; i.e. the hardness in the unprinted regions was 1220 Hv, and, it remains to be 335 Hv in the printed region. The steep change of hardness across the edge of masks reveals that the unprinted regions were nitrided to have much higher hardness than matrix hardness of AISI420. The printed regions were free from infiltration of nitrogen atoms into the matrix.
Figure 9. Transformation of 2D micro patterns to the hardness profile by the present RF-DC plasma N$_2$-H$_2$ for 5400 s at 673 K: a) micro-patterns positive model onto the AISI420 surface after nitriding, b) micro-patterns negative model onto the AISI420 surface after nitriding, c) measured hardness profile along the yellow scanning horizontal line in Figure 9a and 9b.

Figure 10. Diffractogram of RF-DC plasma N$_2$-H$_2$ during 5400 s (optimize of duration), untreated condition, and Bragg position of a) $\alpha'$-Fe, b) $\sigma$-FeCr, c) $\alpha'$-FeN, d) $\varepsilon$-Fe$_2$N, e) $\varepsilon$-Fe$_3$N, and f) CrN.

The powdered diffractogram was analyzed result according to FullProf Suite program [24] of Rietveld [25,26] method. The results of lattice parameters of the crystal structure detailed were presented in Table 3. After [2], no nitrides were synthesized in the lower temperature plasma nitriding; the nitrogen atoms were present as a solute in the Fe/Cr lattices by occupying their vacancy sites.

XRD analysis was performed to investigate whether the iron nitrides were precipitated by RF-DC plasma N$_2$-H$_2$ at 673 K for 5400 s for AISI420 sheet-sample. Hardening mechanism of nitrided AISI420 generally caused by precipitation formation or solid solution atom in matrix specimen. Firstly, precipitation hardening which is usually generated by CrN (37,62°, 43,72°, 63,37°, 76,25°, and 80,31°) precipitation formation in the matrix specimen. Nitrogen atoms diffusing into matrix reacts with chromium in AISI420 [27]. Besides for the original $\alpha'$-Fe (44,76°, 65,16°, and 82,52°) and $\sigma$-FeCr (44,24°) a new peak were detected at $\alpha'$-FeN (82,51°), $\varepsilon$-Fe$_2$N (42,90°, 43,57°, 48,42°, 56,78°, 63,26°, and 67,62°), and $\varepsilon$-Fe$_3$N (30,12°, 49,67°, 57,81°, 73,49°, and 81,72°). Peak was related to the extended martensitic lattice by high nitrogen extraordinary solid solution just as stated in [2] and [28].
Tabel 3. Detailed lattice parameters due to FullProf Suite program [24] of Rietveld [25,26] analysis

| Phase       | Crystal system | Space group | a (Å)     | b (Å)     | c (Å)     | Volume (Å³) | α (°) | β (°) | γ (°) | Ref. |
|-------------|----------------|-------------|-----------|-----------|-----------|-------------|-------|-------|-------|------|
| α'-Fe       | cubic          | Im-3m (229) | 2.86802   | 2.86802   | 2.86802   | 23.590964   | 90    | 90    | 90    | [29] |
| σ-FeCr      | cubic          | Im-3m (229) | 2.90000   | 2.90000   | 2.90000   | 24.38902    | 90    | 90    | 90    | [30] |
| ε-Fe₂N      | trigonal       | P 12 (149)  | 4.79300   | 4.79300   | 4.41700   | 87.876532   | 90    | 90    | 120   | [29] |
| ε-Fe₃N      | trigonal       | P312 (149)  | 4.66800   | 4.66800   | 4.36200   | 82.314819   | 90    | 90    | 120   | [29] |
| CrN         | cubic          | Fm-3m (225) | 4.14800   | 4.14800   | 4.14800   | 71.370078   | 90    | 90    | 90    | [32] |

Figure 11. Schematic picture of the structure obtained using Visualization for Electronic and Structural Analysis (VESTA) v.3.3.2 64 bit [33]: a) α'-Fe (Im-3m), b) σ-FeCr (Im-3m), c) ε-Fe₂N (P312), and d) ε-Fe₃N (P312)

4. Conclusions

Through optimization of the nano-carbon ink contents, the printed patterns have sufficient heat resistivity even during RF-DC plasma in this present works, was found optimal parameters that can be explained in Table 4. Besides for the original α'-Fe and σ-FeCr a new peak were detected at α'-FeN, ε-Fe₂N, and ε-Fe₃N. The hardness on the unprinted surface was 1220 Hv while it remained to be 335 Hv on the printed surface.

Table 4. Summarizes the experimental process parameters for RF-DC plasma N₂-H₂ in this present works

| Process       | Parameters                                                                 |
|---------------|----------------------------------------------------------------------------|
| Pre-sputtering| DC (-450 V), RF (0 V), pressure (70 Pa), temperature (room standard), duration (400 s), and carrier gas (N₂ only) |
| Nitriding     | DC (-300 V), RF (250 V), pressure (70 Pa), temperature (673 K), duration (5400 s), carrier gas (N₂ + H₂) and with partial pressure (N₂ = 100 ml/min; H₂=20 ml/min) |

Acknowledgment

The authors would like to thank Prof. Tatsuhiko Aizawa from Shibaura Institute of Technology (Japan) for the research facilities provided as well as guidance for the implementation of the research in LLC Nanofilm & Coat Laboratory, Ota-ku, Tokyo (Japan). The authors are also thankful to ASMAT Research Collaboration, the University of Brawijaya, which is funded through Program Hibah Kerjasama Luar Negeri JSPS Dikti with the contract number 063/SP2H/LT/DRPM/IV/2017 so that this research can be accomplished.

The authors would like to express gratitude to Dr. E.E. Yunata, Mr. Y. Seki, Mr. A. Farghari, and Mr. S. Kurozumi from Shibaura Institute of Technology (Japan), Dr. K. Wasa from TECDIA, Co. Ltd. (Japan), and Mr. H. Morita from LLC Nanofilm & Coat Laboratory, Ota-ku, Tokyo (Japan) for their help in experiments.
References

[1] Taylan A, Blaine L and Y.C Yen 2001 *J. of Manu. Tech.* **50**(2) 404
[2] Tatsuhiko A, Tatsuya F and Hiroshi M 2016 *J. of Manu. Rev.* **3**(5) 1
[3] I Etsion 2004 *J. of Tribo. Lett.* **17** 733
[4] T Czerwiec, G Marcos, T Thiriet, Y Guo and T Belmonte 2009 *Proc. of IOP Conf. Series: Mater. Sci. & Eng.* **5** 012012-1
[5] Tatsuhiko A, Saya S and Tetsuya Y 2014 *Proc. 9th ICOMM* **15** 26D4-15-p.1
[6] A.V Filippov, S-Y Tarasov, O.A Podgornyh, N.N Shamarin and E.OFilippova 2016 *AIP Conf. Proc.* **1783** 020057-1
[7] Takahisa K, Tatsuhiko A and Tetsuya Y 2015 *J. of Manu. Rev.* **2**(2) 1
[8] Tatsuhiko A, Masahiro T and Tatsuya F 2014 *J. of Procedia Eng.* **81** 1427
[9] Tatsuhiko A and Tetsuya Y 2014 *Proc. 9th IWMF* **12** 55
[10] Tatsuhiko A, Hiroaki S and Tetsuya Y 2015 *Proc. MATEC* **21** 09002-p.1
[11] I Alphonsa, A Chainani, P.M Raole, B Ganguli and P.I John 2002 *J. of Surf. & Coat. Tech.* **150**(2-3) 263
[12] V Valasamudram, S.S.M Nazirudeen, P Chandramohan and K.P Thenmozhi 2008 *J. Anti-Cor. Meth. & Mater.* **55**(2) 73
[13] C.A Figueroa, F Alvarez, D.R.G Mitchell, G.A Collins and K.T Short 2006 *J. Vac. Sci. Technol. A* **24**(5) 1795
[14] Pilch O and Hruby V 2016 *J. of Sol. Sta. Phen.* **258** 395
[15] M.J Baldwin, G,A Collins, M.P Fewell, S.C Haydon, S Kumar, K.T Short and J Tendys 1997 *J. Appl. Phys.* **36** 4941
[16] Yun-tao X, Dao-xin L and Dong H 2008 *J. of Surf. & Coat. Tech.* **202**(12) 2577
[17] A Saeed, A.W Khan, F Jan, H.U Shah, M Abrar, M Zaka-Ul-Islam, M Khalid and M Zakaullah 2014 *J. of Plas. Sci. & Tech.* **16**(5) 462
[18] L Petitjean and A Ricard 1984 *J. of Phys. D: Appl. Phys.* **17** 923
[19] M Hannemann, S Hamann, I Burlacov, K Börner, H-J Spies and J Röpcke 2013 *J. of Surf. & Coat. Tech.* **235** 564
[20] Yoon-Ho C, Ji-Hun K, Kwang-Hyun P, Won-Tae J and Y.S Hwang 2005 *J. of Surf. & Coat. Tech.* **193** 321
[21] K.S Suraj, P Bharathi, V Prahlad and S Mukherjee 2007 *J. of Surf. & Coat. Tech.* **202** 303
[22] Istroyah, I.N.G Wardana and Dionysius J.D.H.S 2014 *J. of App. Mech. & Mater.* **493** 755
[23] M.J Baldwin, G.A Collins, M.P Fewell, S.C Haydon, S Kumar, K.T Short and J Tendys *Jpn. J. of Appl. Phys.* **36** 4941
[24] Juan R-C 1993 *J. of Phys. B* **192** 58
[25] Vikash K, Swati K, Pawan K, Manoranjan K and Lawrence K 2015 *J. of Adv. Mater. Lett.* **6** 141
[26] A.L Ortiz, F.L Cumberera, F Sanchez-Bajo, F Guibert EA and R Caruso 2000 *J. of the Euro. Cera. Soci.* **20** 1846
[27] Istroyah, I.N.G Wardana and Dionysius J.D.H.S 2016 *J. of App. Mech. & Mater.* **836** 214
[28] Abdelrahman F and Tatsuhiko A 2017 *J. of Mater. Trans.* **58**(4) 699
[29] S.B Hendricks and P.R Kosting 1930 *Int. J. of Crys. Geo. Crys. Phys. Chem. Crys.* **74** 520
[30] T.I Badjuk, G.P Kushma and O.I Rybachio 1974 *Proc. of Chern. Metal.* **17** 126
[31] Jette O, Thomas L.C, Kenny S and Marcel A.J.S 2008 *J. of Mater. Sci.* **43** 5358
[32] M.N Eddine, E.F Bertaut, M Roubin and J Paris 1977 *J. of Acta Crys. B* **33** 3010
[33] Koichi M and Fujio I 2011 *J. of App. Crys.* **44** 1272