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Study of Nano Mechanical and Nano Structure on Titanium Nitride (TIN) Coating Prepared by RF Magnetron Sputtering

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Abstract--- *Titanium nitride (TiN) coating is one in every of the foremost used thin films. TiN coating ready on low carbon steel substrate by dc Magnetron sputtering. Its smart producing sturdiness and low value. The structure and properties can characterization X-ray diffraction (XRD), scanning Electron microscopy (SEM), nano indentation and tribological. The small structural feature and corrosion performance are going to be compared, the layers of coating and enhance the corrosion performance of periodical vapor deposition PVD TiN coated steel. The result can expect to point the entire were rate may well be scale back by half with relevance steel once this type of coating is employed. The liquid corrosion behavior of the chemical compound coating (TiN) is powerfully passionate about the micro defect density of the coating.*

Keywords--- *RF /DC Magnetron Sputtering, Corrosion, Titanium nitride (TiN), X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Nano Indentation and Tribological.*

I. INTRODUCTION

Currently an ample and diverse range of materials can be used for manufacturing a wide spread series of applications in order to meet consumer needs. Titanium nitride is utilized in a wide range of applications for space, biomedicine, and microelectronics industry and so on due to its excellent physical, chemical, electrical and mechanical properties. The properties that make titanium nitride suitable for application on worm gear are its hardness, good adhesive wear, and resistance to corrosion. Tin layer is deposited by R.f magnetron sputtering using N2 atmosphere. Submission Cover Sheet, a Discussion Submission Cover Sheet, and an IEEE Copyright Form.

II. RESEARCH METHODOLOGY

X-Ray Powder Diffraction: X-ray powder diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material is finely ground, homogenized, and average bulk composition is determined.

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Max von Laue, in 1912, discovered that crystalline substances act as three-dimensional diffraction gratings for X-ray wavelengths similar to the spacing of planes in a crystal lattice. X-ray diffraction is now a common technique for the study of crystal structures and atomic spacing.X-ray diffraction is based on constructive interference of monochromatic X-rays and a crystalline sample. These X-rays are generated by a cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate, and directed toward the sample. The interaction of the incident rays with the sample produces constructive interference (and a diffracted ray) when conditions satisfy Bragg's Law ($n\lambda=2d \sin \theta$). This law relates the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample. These diffracted X-rays are then detected, processed and counted. By scanning the sample through a range of 2θangles, all possible diffraction directions of the lattice should be attained due to the random orientation of the powdered material. Conversion of the diffraction peaks to d-spacings allows identification of the mineral because each mineral has a set of unique d-spacings. Typically, this is achieved by comparison of d-spacings with standard reference patterns.

Atomic Force Microscopy: The atomic force microscope (AFM) is one kind of scanning probe microscopes (SPM). SPMs are designed to measure local properties, such as height, friction, magnetism, with a probe. To acquire an image, the SPM raster-scans the probe over a small area of the sample, measuring the local property simultaneously. AFMs operate by measuring force between a probe and the sample. Normally, the probe is a sharp tip, which is a 3-6 um tall pyramid with 15-40nm end radius. Though the lateral resolution of AFM is low (~30nm) due to the convolution, the vertical resolution can be up to 0.1nm.To acquire the image resolution, AFMs can generally measure the vertical and lateral deflections of the cantilever by using the optical lever. The optical lever operates by reflecting a laser beam off the cantilever. The reflected laser beam strikes a position-sensitive photo-detector consisting of four-segment photo-detector. The differences between the segments of photo-detector of signals indicate the position of the laser spot on the detector and thus the angular deflections of the cantilever. Piezo-ceramics position the tip with high resolution. Piezoelectric ceramics are a class of materials that expand or contract when in the presence of a voltage gradient. Piezo-ceramics make it possible to create three-dimensional positioning devices of arbitrarily high precision. In contact mode, AFMs use feedback to regulate the force on the sample. The AFM not only measures the force on the sample but also regulates it, allowing acquisition of images at very low forces. The feedback loop consists of the tube scanner that controls the height of the tip; the cantilever and optical lever, which measures the local height of the sample; and a feedback circuit that attempts to keep the cantilever deflection constant by adjusting the voltage applied to the scanner. A well-constructed feedback loop is essential to microscope performance

Salt Spray Test: This accelerated laboratory test was invented at the beginning of the 20th century. It provides a controlled corrosive environment and has been used to produce relative corrosion-resistance information for specimens of metals and coated metals exposed in a test chamber. The classical salt spray (fog) test ASTM B117 consists of atomizing a salt solution into uniform droplets on specimens supported or suspended between 15-30°from the vertical. The salt solution is a solution of 5% (in weight) of NaCl, (more than sea water, which is only 1.8% to max 3%). The exposure zone of the salt spray chamber is maintained at 35°C.The pH of the salt solution is such that when atomized at 35°C, the collected solution will be in a pH range from 6.5 to 7.2.The test is continuous for the duration of the entire test period. The period of exposure is mutually agreed upon between the purchaser and the seller. It can reach more than 1000H.There exist other accelerated testing procedures – in ageing tests, quite often used in automotive industry. These tests are briefly described below. The most important corrosive element is moisture, which is applied in all ageing tests, supplemented by salt mist and/or changing temperature.

Scratch Test: Figure shows Ti interlayer thickness effect in the intrinsic stress and the adhesion (critical load L_c) of the deposited system. The film without interlayer presented higher dispersion caused by an irregular substrate deformation, generated by the film stress. As the interlayer thickness is increased, a decrease in the intrinsic stress was observed because Ti film supports the Ti and N atoms or TiN molecule bombardment, presenting plastic deformation and avoiding perturbations on the TiN lattice .Moreover, the role of the Ti interlayer is to dissolve any oxide layer remaining on the surface of the substrate as well as to relieve shear stress in the interface. Results also show a decrease in the stress up to a certain interlayer thickness value. Nevertheless, after this value, the stress tends to be stable. This indicates that there is a critical Ti interlayer thickness for effectively relieving residual stress. The scratch test was performed in order to study the film's adhesion. Figures show an increase on the critical load as the Ti interlayer thickness increases. Due to the TiN film interdiffusion increment, intrinsic stress diminution and total thickness increase, the load capability is enhanced. This behavior avoids film delamination; nevertheless, it has been found that higher-thickness ceramic films can reduce adhesion due to a high dislocation density on the interface. For thicker films, not only the delamination is avoided, but also the spalling degree becomes lower as the load is increased. This demonstrates that the increase of film thickness from a certain critical value can improve film adhesion. This type of failure is called bulking spallation, which is presented in the form of irregular arcs on the track. These failures are generally presented in hard films deposited on ductile substrates .Hardness and elastic moduli plots versus penetration depth were obtained by nano indentation.

III. RESULT AND DISCUSSIONS

Structure and Element Distributions: The phase composition and the structure of the film were studied by X-Ray diffraction analysis. The XRD patterns of TiN thin films are shown in the figure 6.1.The excellent peaks (111), (200) and (311) were obtained in the power X-Ray diffraction studies. The peaks were compared with Origin 8 and Match! Diffraction patterns from the observed peaks corresponding to the formation of tetragonal phase of TiN were indexed according to tetragonal structure. Knowing the wavelength (**λ),** full width at half maximum (FWHM) of the peaks (β) and diffraction angle (Θ), the particle size (D) was calculated using the Scherrer formula.

$$D = 0.9 \lambda / \beta \cos\Theta$$

From the relation, the average sized TiN was determined to be 8.3nm.

AFM Characterization: Surface topographical characterization was done my Atomic Force Microscopy. The AFM scan was carried on three samples coated with TiN at room temperature, 400C and 600C for 40nm, 60nm and 40nm respectively. Scan was carried with semi-contact mode on sputtered TiN for a scan area of 5μm × 5μm on the surface. From the AFM images (refer Fig.6.6, Fig 6.8, and Fig 6.10) titanium surface have average roughness of

55.6423 nm, 42.3304 nm and 23.1762 nm for room temperature, 400°C and 600°C temperature coatings respectively. From the results found it can be determined that due to the low average roughness, there will be low friction co-efficient decreasing the wear on the worm gear.

Salt Spray Test: The samples coated with TiN where under salts spray test for 12 hours. The concentration of sodium chloride was 5.2% NaCl and the temperature in the chamber was 34.1°C to 35.6°C. The pH of the salt solution was 6.9 and air pressure was 15 psi. After 12 hours of salt spray test it was observed that there was no corrosion. From the data acquired, it can be inferred that the corrosion resistance of low carbon steel has been increased.

Scratch Test: The scratch test was performed in order to study the film's adhesion. Results show an increase on the critical load as the Ti interlayer thickness increases.

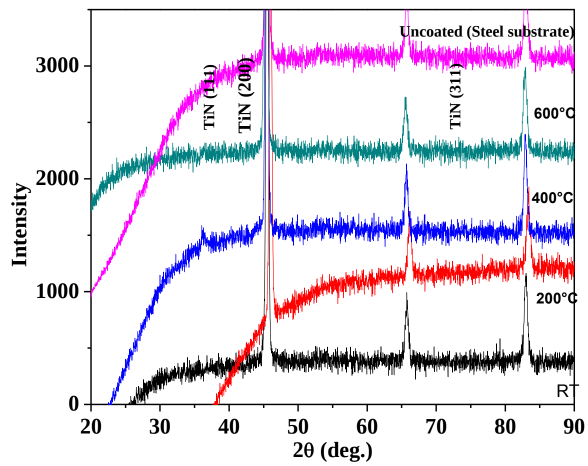

Fig.6.1: XRD Comparision Graph

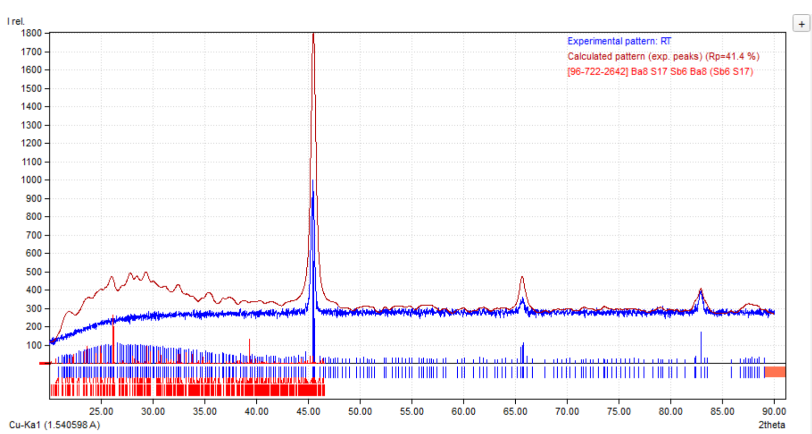

Fig 6.2: XRD – Results for Room Temperature Coated Piece

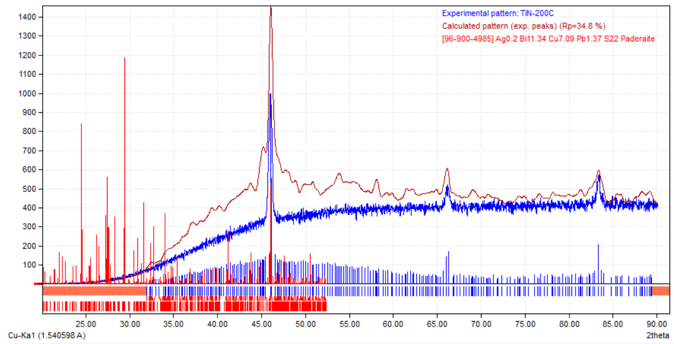

Fig 6.3: XRD – Results for 200°C Coated Pieces

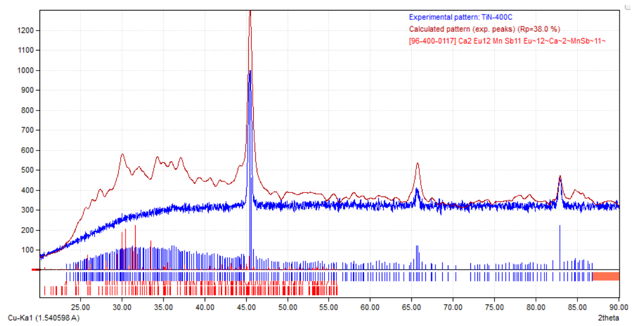

Fig 6.4: XRD – Results for 400°C Coated Peices

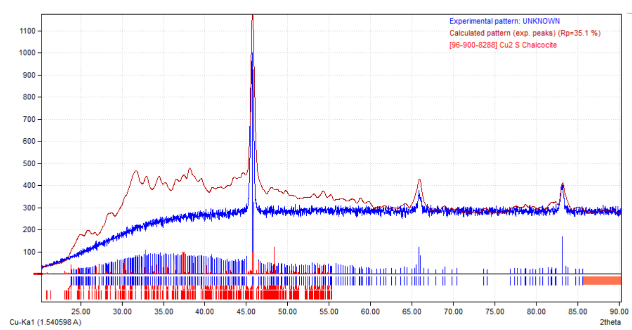

Fig 6.5: XRD – Results for 600°C Coated Pieces

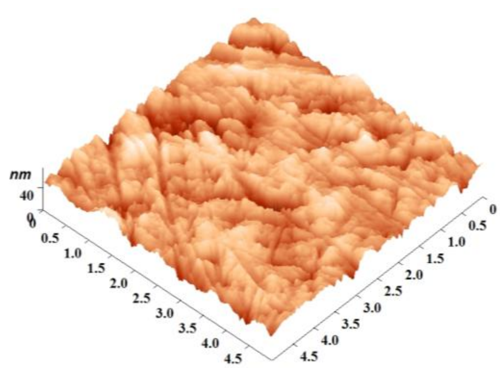

Fig 6.6: 5µm × 5µm 3D Image Of Coated Sample At Room Temperature

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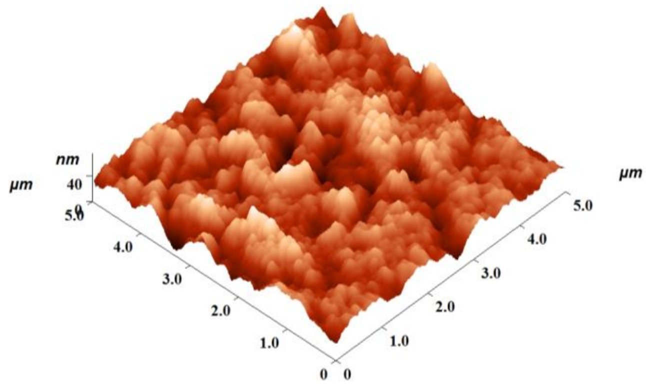


Fig 6.7: 5μm × 5μm 3D Image of Sample Coated At 400°C Temperature

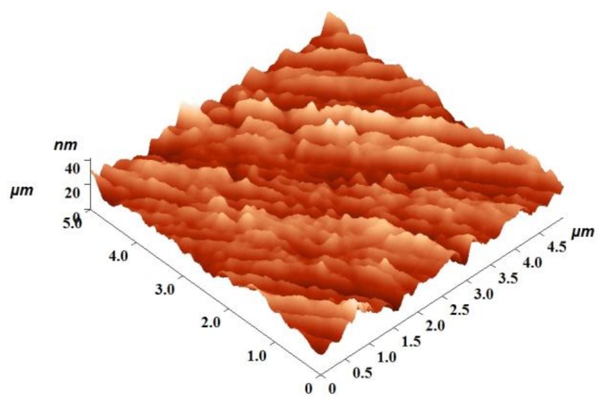
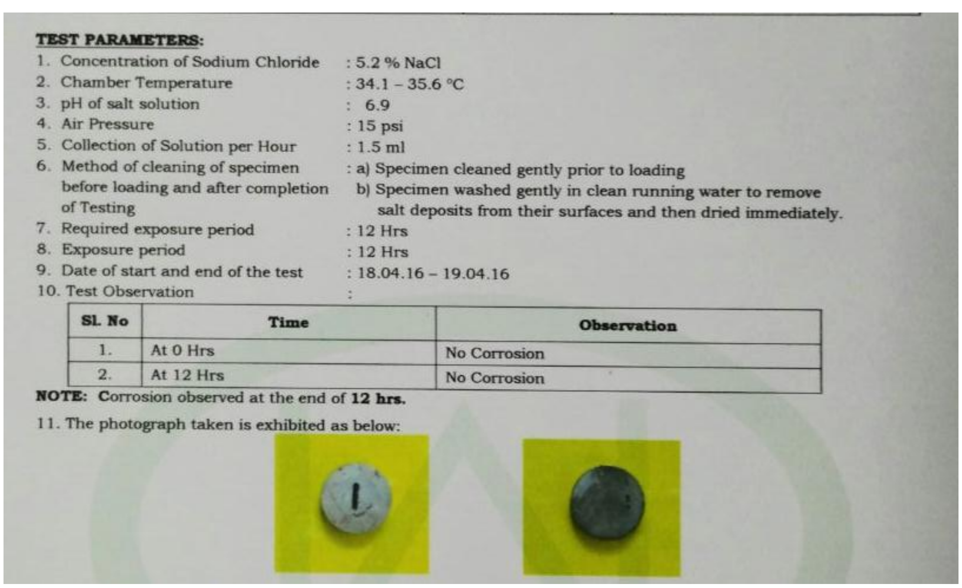


Fig 6.8: 5μm × 5μm 3D Image of Coated Sample At 600°C Temperature



TEST PARAMETERS:

1. Concentration of Sodium Chloride : 5.2 % NaCl
2. Chamber Temperature : 34.1 – 35.6 °C
3. pH of salt solution : 6.9
4. Air Pressure : 15 psi
5. Collection of Solution per Hour : 1.5 ml
6. Method of cleaning of specimen before loading and after completion of Testing : a) Specimen cleaned gently prior to loading
   b) Specimen washed gently in clean running water to remove salt deposits from their surfaces and then dried immediately.
7. Required exposure period : 12 Hrs
8. Exposure period : 12 Hrs
9. Date of start and end of the test : 18.04.16 – 19.04.16
10. Test Observation :

Sl. No	Time	Observation
1.	At 0 Hrs	No Corrosion
2.	At 12 Hrs	No Corrosion

NOTE: Corrosion observed at the end of **12 hrs.**

11. The photograph taken is exhibited as below:

Fig 6.9: Salt Spray Test Results for Room Temperature Coated Sample

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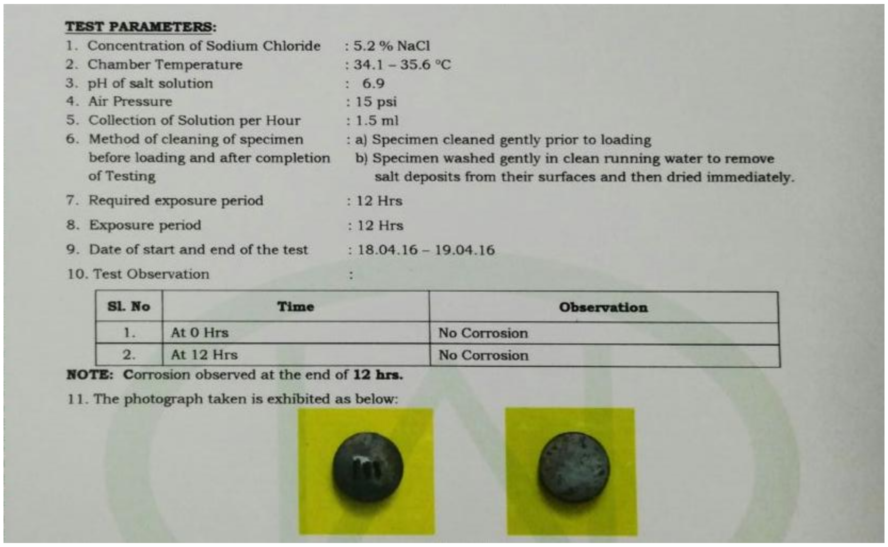


TEST PARAMETERS:

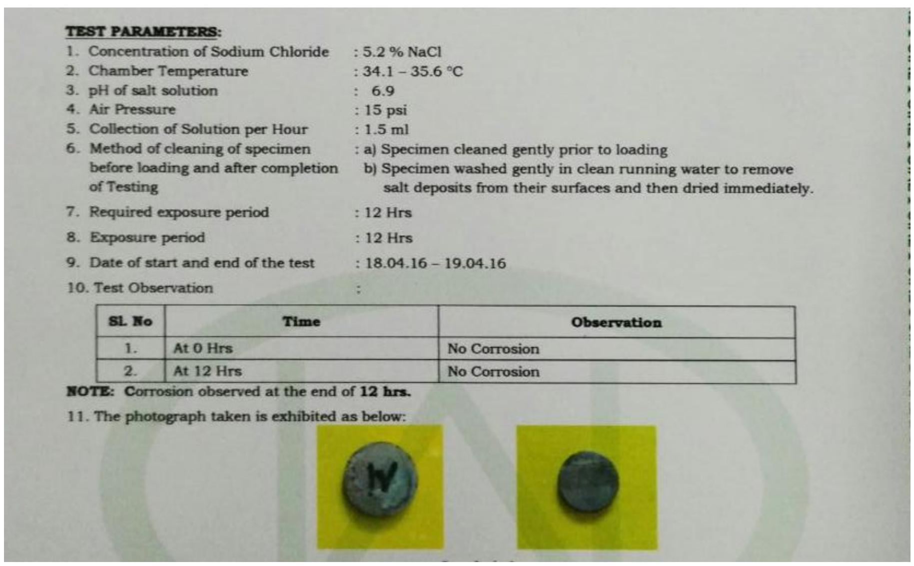
1. Concentration of Sodium Chloride : 5.2 % NaCl
2. Chamber Temperature : 34.1 – 35.6 °C
3. pH of salt solution : 6.9
4. Air Pressure : 15 psi
5. Collection of Solution per Hour : 1.5 ml
6. Method of cleaning of specimen before loading and after completion of Testing : a) Specimen cleaned gently prior to loading b) Specimen washed gently in clean running water to remove salt deposits from their surfaces and then dried immediately.
7. Required exposure period : 12 Hrs
8. Exposure period : 12 Hrs
9. Date of start and end of the test : 18.04.16 – 19.04.16
10. Test Observation :

Sl. No	Time	Observation
1.	At 0 Hrs	No Corrosion
2.	At 12 Hrs	No Corrosion

NOTE: Corrosion observed at the end of **12 hrs.**

11. The photograph taken is exhibited as below:

Fig 6.10: Salt Spray Test Results for 400°C Temperature Coated Sample



TEST PARAMETERS:

1. Concentration of Sodium Chloride : 5.2 % NaCl
2. Chamber Temperature : 34.1 – 35.6 °C
3. pH of salt solution : 6.9
4. Air Pressure : 15 psi
5. Collection of Solution per Hour : 1.5 ml
6. Method of cleaning of specimen before loading and after completion of Testing : a) Specimen cleaned gently prior to loading b) Specimen washed gently in clean running water to remove salt deposits from their surfaces and then dried immediately.
7. Required exposure period : 12 Hrs
8. Exposure period : 12 Hrs
9. Date of start and end of the test : 18.04.16 – 19.04.16
10. Test Observation :

Sl. No	Time	Observation
1.	At 0 Hrs	No Corrosion
2.	At 12 Hrs	No Corrosion

NOTE: Corrosion observed at the end of **12 hrs.**

11. The photograph taken is exhibited as below:

Fig 6.11: Salt Spray Test Results for 600°C Temperature Coated Sample

IV. CONCLUSION

TiN thin films are prepared by PVD methods. Initial characterization of films by AFM and XRD was deposited. TiN coatings were with successfully prepared RF magnetron sputtering on low steel substrate. TiN coatings might achieve higher corrosion polarization resistance and comparatively stable corrosion potential within the SBF surroundings than the uncoated low steel. Therefore, the coated samples would have a lower corrosion and therefore the substrate coated at 400°C exhibited the most effective corrosion resistance for the coating investigated within the studies. The damage resistance and therefore the corrosion resistance of low steel was multiplied by TiN coating.

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