



Journal of Metallic Material Research

















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ARTICLE Structure, Adhesion Strength and Corrosion Resistance of Vacuum Arc Multi-Period NbN/Cu Coatings

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ARTICLE INFO	ABSTRACT
Article history Received: 1 November 2019 Accepted: 14 November 2019 Published Online: 31 March 2020	The influence of deposition modes on the phase-structural state, corrosion resistance, and adhesive strength of vacuum-arc multi-period NbN/Cu coatings is studied. It was found that in thin layers (about 8 nm, in a constant rotation mode), regardless of the change in the pressure of the nitrogen atmosphere, a metastable δ - NbN phase forms (cubic crystal lattice of the
Keywords: Vacuum Arc method NbN/Cu Phase composition Adhesion strength Impedance spectroscopy Polarization resistance	NaCl type). At a layer thickness of ~ 40 nm or more, a phase composition changes from the metastable δ - NbN to the equilibrium ε - NbN phase with a hexagonal crystal lattice. In the presence of the ε - NbN phase in the niobium nitride layers, the highest adhesive strength is achieved with a val- ue of LC5 = 96.5 N. Corrosion resistance tests have shown that for all the studied samples the corrosion process has mainly an anodic reaction. The highest corrosion resistance was shown by coatings obtained at a pressure of 7 $\cdot 10^4$ Torr, with the smallest bias potential of -50 V and the smallest layer thickness; with a thickness of such a coating of about 10 microns, its service life in the environment of the formation of chloride ions is about a year.

1. Introduction

N iobium nitride (NbN) has a high melting point (about 2600 K), which is determined by the high binding energy (14.81 eV)^[1]. NbN-based coatings have high functional characteristics (high hardness, wear resistance ^[2] and oxidation resistance ^[3], etc.), and also have a high critical temperature of superconductivity (Tc \approx 16 K)^[4]. There are several phases in the Nb-N system: β , δ , ε , γ , δ' , and η ^[5], which allows one to create different phase-structural states of coatings from niobium nitrides. Moreover, as is known, for nonequilibrium formation conditions (using vacuum-arc methods) for transition metal mononitrides, modifications with the structural

type of NaCl are most often stabilized ^[6,7].

It is known that the properties of thin MeN films can be improved by adding a second "immiscible" metal (where Me is a transition metal) ^[8]. As an immiscible metal, Cu and Ag are used. In such a combination (MeCu and MeAg) materials have a good prospect for use as coatings in medical technology. Therefore, it is very important to know the adhesive strength and corrosion resistance of such coatings, which this work is devoted to ^[9].

The second important goal of this work was to establish the relationship of the deposition parameters with the phase-structural state and properties. This is the basis of structural engineering ^[10] and allows one to achieve the

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necessary properties by modeling the structure.

2. Material and Methods

The coatings were deposited on AISI 321 austenitic steel by the vacuum-arc method at the "Bulat-6" installation with various technological deposition conditions, the modes of which are given in Table 1.

Table 1. Technological parameters of deposition	of NbN/
Cu coatings	

Series	PN, Torr	Ub, V	The condition for obtaining	Layer Thickness	
1	7.10-4	-50	constant rotation	of about 8 nm	
2	3.10-3	-50	constant rotation		
3	7.10-4	-50	20 a interval	40 nm	
4	7.10-4	-200	20 S Intervar	40 1111	
5	3.10-3	-100	120 s – Cu 300 s – Nb	240 nm – Cu 600 nm – Nb	

Working gas pressure (PN) during coating deposition was $7 \cdot 10$ -4 or $3 \cdot 10$ -3 Torr; the bias potential supplied to the substrate varied from -50 to -200 V. The deposition was carried out from 2 sources (Nb and Cu) in the regimes with a constant rotation speed (rotation speed of 8 rpm) and in discrete mode (with a stop for 20 seconds near each of the plasma sources or with a stop of 120 s and 300 s for Cu and Nb layers, respectively). The total coating time was 1.5 hours. The thickness of the coatings was about 12 μ m.

The phase-structural state of the samples was studied by X-ray diffractometry using a DRON-4 apparatus in Cu-K α radiation. To monochromatize the detected radiation, a graphite monochromator installed in the secondary beam (in front of the detector) was used. To decode the diffraction patterns, tables of the Powder Diffraction File international center were used ^[11]. The separation of profiles into components was carried out using the NewProfile software package (developed by NTU KhPI, Ukraine).

The surface morphology of the coated samples was studied using optical and scanning electron microscopy (SEM) on ZEISS AXIO Ver A1 instruments and FEI Nova NanoSEM 450, respectively.

The coatings were examined for their corrosion resistance during electrochemical processes. Electrochemical tests were carried out using a 3-electrode cell with a capacity of 200 ml and a Biologic SP-150 potentiostat. The cell consists of a coated sample (working electrode), a saturated calomel electrode (reference electrode), and a platinum electrode (counter electrode). Corrosion resistance was assessed by measuring the open-circuit potential for 1.5 hours in a solution of 0.9% NaCl at room temperature. Impedance spectroscopy was performed in the frequency range from 10^{-2} to 10^{5} Hz. The potentiodynamic polarization test was carried out in the range from -0.6 to +1 V at a scan speed of 1 mV/s. The contact area of the sample with the electrolyte was 0.196 cm².

Determination of adhesive and cohesive strength, resistance to scratching and elucidation of the mechanism of destruction of coatings was carried out using a scratch tester Revetest (CSM Instruments). The contact load was 0.9 N, and the loading speed was 5 N/s.

3. Results and Discussion

The study of surface morphology showed that for coatings at a pressure of $PN = 7 \cdot 10^4$ Torr, the bias potential increases from -50V (Figure 1 a) to -100V (Figure 1 e) and - 200V (Figure 1 g) leads to a decrease in the number and size of the droplet phase. An increase in pressure from $7 \cdot 10$ -4 to $3 \cdot 10$ - 3 Torr (Figure 1 b) also leads to a decrease in the amount of the drop phase.



h





The energy dispersion spectra and elemental composition of the studied coatings are shown in Figure 2.





a - series 1, b - series 2, c - series 3, d - series 4, e - series 5

Figure 2. Energy dispersive spectra with data on elemental composition

It can be seen that for series 1 and 2 obtained in the constant rotation mode, the elemental composition remains almost unchanged with increasing pressure of the nitrogen atmosphere. Series 3 and 4, obtained at a pressure of PN = $7 \cdot 10$ -4 Torr in the discrete mode, have a noticeable difference: with an increase in the bias potential from -50 V to -200 V, the amount of copper decreases by 2 times.

The study of the phase-structural state was carried out using the XRD method. The X-ray diffraction spectra of the coatings are shown in Figure 3.



Figure 3. X-ray diffraction spectra of NbN/Cu multilayer coatings

It can be seen that at a relatively low pressure PN = $7 \cdot 10^{-4}$ Torr (spectrum 1, Figure 3) in the constant rotation mode, two phases are formed with an fcc crystal lattice (structural type NaCl): metastable δ -NbN (JCPDS 38-1155) and Cu (JCPDS 89-2838). The peak ratio is close to the standard for the fcc lattice; therefore, no pronounced texture is observed. An increase in pressure to $3 \cdot 10$ -3 Torr (spectrum 2, Figure 3) leads to

a qualitative change in diffraction spectra. In addition to the formation of the axis of preferential orientation ^[11], a diffraction peak is revealed from the equilibrium ϵ -NbN (JCPDS 89-4757) phase with a hexagonal lattice.

An increase in the layer thickness to 40 nm (spectra 3, 4, Figure 3) leads to the formation of only the equilibrium ϵ -NbN phase and Cu. An increase in the bias potential to Ub = -200 V (spectrum 4, Figure 3) does not lead to a change in the phase composition. However, in this case, the preferred orientation of crystallites with the (004) plane perpendicular to the growth surface is formed.

At the largest layer thickness (about 240 nm for Cu and 600 nm for NbN), a complete spectrum of diffraction peaks of ϵ -NbN and Cu phases is formed without a notice-able preferential orientation (spectrum 5, Figure 3).

Corrosion testing allowed us to determine the potential (Ecorr) and current (Icorr) of corrosion by extrapolation using the Tafel method. Figure 4 presents potentiodynamic polarization curves (graphs of the dependence of the potential on the density of the logarithm).



1 – series 1, 2 – series 2, 3 – series 3, 4 – series 4, 5 – series 5

Figure 4. Potentiodynamic polarization curves

As can be seen from Figure 4, for all series of coatings, the process is controlled by the anode part of the polarization curves ^[12]. From the above curves, the slope coefficients of the linear sections of the anode and cathode parts βc and βa were obtained (Table 2).

The corrosion rate is proportional to the corrosion current and was calculated by the formula

$$CR = \frac{I_{corr} \cdot K \cdot EW}{d \cdot A},$$
(1)

where CR is the corrosion rate, mm/year; Icorr - corrosion current, mA; K is the conversion factor determining the unit of measurement of the corrosion rate; EW - equivalent weight, gram- equivalent; d is the density, g/cm³; A is the sample area, cm².

Additionally, the polarization resistance (Rp) of the Tafel curves was calculated using the Stern-Geary equation ^[13], which takes into account both the current density and the slope of the polarization curves.

The calculation results are given in table 2.

Se- ries	Ecorr, mV	Icorr, mA	βa, mV	βc, mV	CR, mm/year	Rp, Ohm∙cm²
1	-208,347	9·10 ⁻⁵	119,2	103,6	8,14·10 ⁻³	5252,8
2	-138,302	4,21.10	104,2	187,5	38·10 ⁻³	13563,2
3	-258,219	1,17·10 ⁻	181,7	290,0	106.10-3	8094,8
4	-303,715	1,96·10 ⁻ 3	2155,7	286,3	176.10-3	10976
5	-174,249	1,48.10	83,4	156,5	134.10-3	2352

Table 2. Potentiodynamic polarization test results

As can be seen from the table 2, all coatings have good corrosion resistance, regardless of the coating conditions (table 1). However, the results show that the application mode and the number of layers change the resistance of the coating in an aggressive environment. So, the best indicators of corrosion resistance are characteristic of coatings of series 1 and 2 obtained in the continuous deposition mode (when the layer thickness is minimal and about 8 nm), as well as with the smallest bias potential (Ub = -50V). Which may be explained by the lower defective-ness of thin layers.



Figure 5. Curves of corrosion potential over time

As can be seen from the curves in Figure 5, during the interaction of the coating with the electrolyte medium, the surface layer slowly dissolves with a slight manifestation of pitting. This situation is typical for the series of samples 1, 3, 4. To a greater extent, the same trend is observed for series 5 (except for pitting formation). The behaviour of the coating of series 2 is radically different. At the initial time, the corrosion potential is relatively large, but it decreases with a tendency to stabilize.

The resistance of charge transfer through protective coatings using Nyquist curves was also evaluated. The curves themselves for the systems under consideration and the equivalent circuit model are shown in Figure 6.

Based on the conducted corrosion tests, the best indicators of corrosion resistance were shown by coatings of series 1 and 2. Additionally, studies were carried out for these 2 series, which allow us to determine one of the most important characteristics of the mechanical properties of coatings - adhesive strength. To determine it, the method of scratch testing was used.



C1, C2 - the capacity of the double layer and coating, respectively; R1, R2 - resistance of the electrolyte and phases in the coating, respectively 1 - series 1, 2 - series 2, 3 - series 3, 4 - series 4, 5 - series 5

Figure 6. Nyquist curves and equivalent electrolyte-coating circuit model

Figure 7 shows the wear paths in the region of LC critical points during loading. The resulting critical point values for these areas are given in table 3. LC1







Figure 7. Wear paths at critical points under LC loading for coatings of series 1 (a) and 2 (b)

Cowing	The value of the load in the region of critical points, N				
Series	LC1	LC2	LC3	LC4	LC5
1	23.6	29.5	34.3	38.6	61.8
2	23.8	30.3	35.3	40.1	96.5

 Table 3. The magnitude of the load in the region of critical points LC

It can be seen that in the area of primary crack formation and up to the critical point LC4, coatings of series 1 and 2 have almost the same values. However, the critical point LC5 (the load at which complete abrasion of the coating occurs) is higher for series 2. This can be attributed to a change in the phase-structural state and the appearance of the ε -NbN phase, as well as a significant decrease in the droplet phase on the coating surface.

4. Conclusions

Studies have shown that the technological parameters of deposition have a significant impact on the phase-structural state, corrosion resistance and adhesive strength. An optimal deposition mode has been established to obtain the best corrosion resistance indicators: a bias potential of -50 V at the smallest layer thickness of ~ 8 nm. In this mode, a metastable phase of δ -NbN and Cu with a face-centered crystal lattice is formed. Comparison of coatings of series 1 and 2 (showing the best values of corrosion resistance) on adhesive strength show that higher adhesion strength indications for series 2 (critical load LC5 = 96.524 N). Based on the data obtained, it is clear that there is no universal structural state that provides both the highest adhesive strength and corrosion resistance. However, the necessary functional properties can be achieved by structural engineering at the stage of coating formation. In this work, it was found that the complex of the highest functional characteristics is characteristic of coatings of series 1. They were obtained at a pressure of 7.10-4 Torr, a bias potential of -50 V, a layer thickness of 8 nm and have a δ -NbN phase in niobium layers (no pronounced texture is observed). These coatings have the best indicators of corrosion resistance among the investigated series of coatings, while also quite high values of adhesive strength. The assessment showed that with a thickness of such a coating of about 10 microns, the resource of its operation in an aggressive environment is about a year.

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REVIEW Research Progress of Superhydrophobic Polymer Composite Coatings for os Magnesium Alloys

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ARTICLE INFO	ABSTRACT
Article history Received: 16 January 2020 Accepted: 14 March 2020 Published Online: 31 March 2020	Magnesium (Mg) alloy is the lightest metal material found because of its excellent physical and mechanical properties, specific strength, biocompatibility and biomechanical compatibility, therefore, it has very promising development prospects in aerospace, automobile manufacturing, and biodegradable materials. However, due to the relatively chemical properties
<i>Keywords:</i> Superhydrophobic Polymer Nanoparticle	of magnesium alloys, poor corrosion resistance, fast degradation rate, and poor wear resistance, they have been greatly restricted in practical applica- tions. Therefore, anti-corrosion measures of magnesium alloys are particu- larly important. The manufacture of hydrophobic surfaces is a very effec- tive method of anti-corrosion. The surface of super-hydrophobic polymer composites (i.e., thin coatings) is constructed on the surface of magnesium alloy materials to enhance their corrosion resistance and wear resistance, and the effect of its articertic measures is not impressing.

1. Introduction

Agnesium (Mg) alloy has attracted worldwide attention due to its excellent mechanical properties, high specific strength and specific stiffness, low density, good electromagnetic shielding, and excellent biocompatibility ^[1-3], which has become the most remarkable green environmental protection engineering material in the 21st century. In recent years, it has been favored in aerospace, automobile manufacturing, industrial materials, biodegradable materials and medical materials, and has achieved excellent research results. However, the poor wear resistance & corrosion resistance and fast degradation rate of magnesium alloys ^[4,5], make magnesium alloys greatly hindered in practical applications. This rapid and uncontrollable rate of corrosion or degradation significantly reduces the mechanical strength of the magnesium alloy, causes the material to fail prematurely, and the service life cannot reach the expected effect. In biodegradable materials, magnesium alloy releases a large amount of hydrogen due to corrosion, which poses a great threat to the health of patients implanted with materials ^[6]. Therefore, measures to control the corrosion rate or degradation rate of magnesium alloys are particularly important. A protective coating is applied on the surface of the magnesium alloy as a physical barrier to reduce or block the contact between the magnesium alloy and the external corrosive medium, thereby achieving the purpose of protecting the magnesium alloy. Because the super-hydrophobic composite material has the advantages of self-cleaning, drag reduction, good thermal stability, etc., and makes the magnesium alloy anti-corrosion effect more obvious, and the service life is extended, regardless of corrosion resistance, wear resistance, adhesion and

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mechanical properties Get promoted. Therefore, in recent years, superhydrophobic polymer composites have received much attention in the corrosion control of magnesium alloys. This paper mainly introduces the different preparation methods, corrosion resistance and thermal stability of superhydrophobic surfaces, reviews the superhydrophobic polymer composite coatings in recent years, and prospects the development direction of magnesium alloy superhydrophobic composites.

2. Preparation Method of Super-Hydrophobic Surface

At present, the most widely used method for magnesium alloy corrosion control is to prepare superhydrophobic surfaces on the base material to achieve the purpose of corrosion control. There are two common methods for preparing superhydrophobic surfaces ^[7]: the first is to construct micro-nano rough structures on low surface energy materials with a certain hydrophobicity. The second is mainly to modify the surface of micro-nanostructures with substances with low surface energy. The surface of the magnesium alloy base material is hydrophilic, so it is necessary to build a micro-nano structure on the surface, and then modify it with a substance with low surface energy. Common micro-nano structure surface treatment technologies include: chemical etching, hydrothermal method, electrochemical deposition, oxidation method, sol-gel method and vapor deposition. Currently, the surface modification technology using low surface energy materials has been very mature. The main low surface energy materials are: fluoride ^[8], organ silicide ^[9], long-chain alkanes ^[10] and other substances.

2.1 Chemical Etching Method

The chemical etching method is mainly a chemical reaction between the etching solution and the pre-etched material to form a micro-nano structure on the surface of the material, thereby increasing roughness and enhancing the hydrophobic effect. Liu [11] et al. Chemically etched magnesium alloys in silver nitrate solutions of different concentrations for a short period of time, and finally the surface of the magnesium alloy formed crater-shaped and petal-shaped binary micro-nano structures hydrophobic surfaces. A super-hydrophobic surface is obtained by a surface modification treatment with a low surface energy organ silicide. The contact angle is 138.4 °, and the rolling angle is 2 °. Its super-hydrophobic surface has good corrosion resistance. Wang ^[12] et al. Treated the treated magnesium alloy in a salt spray box for 120 min, and then used a low surface energy 1wt.% Fluor silane to perform surface hydrophobic modification to reduce the surface energy. The obtained super-hydrophobic surface generated a micro-nano structure similar to a petal structure. The surface morphology is shown in Figure 1 with a contact angle of 152.65° and a roll angle of 5° . After testing, superhydrophobic surfaces have very good mechanical properties and corrosion resistance.



Figure 1. SEM images of super-hydrophobic surface prepared by chemical etching at different magnifications ^[12]

2.2 Hydrothermal Method

In the hydrothermal method, the reactants are placed in an autoclave aqueous solution, and the hydrothermal reaction is performed under high temperature and high-pressure conditions. The advantage of this method is that it can well control the crystal form and has good dispersibility. Zhang ^[13] et al. Used hydrothermal treatment and then awakened the surface with hydrophobic modification by stearic acid to finally produce a superhydrophobic surface with a surface shape similar to a petal structure with a static contact angle of 157.6 °. And the prepared super-hydrophobic coating significantly improves the corrosion resistance of the magnesium alloy. Zheng ^[14] et al. used a simple one-step hydrothermal method to prepare a hydrophobic coating on the surface of a magnesium alloy, and surface modified with stearic acid. The resulting surface has a superhydrophobic function and a contact angle of about 146 °. Corrosion behavior of the prepared magnesium alloy sample in a 3.5 wt% NaCl solution was compared with the untreated sample, and a great improvement in corrosion resistance was found.

2.3 Electrochemical Deposition Method

The electrochemical deposition method refers to a technology of forming a coating layer by an oxidation-reduction reaction occurring on an electrode through the migration of positive and negative ions in an electrolyte solution under the action of an external electric field. Liu ^[15] et al.

formed a cauliflower-like clustered micro-nanostructured superhydrophobic surface on a magnesium alloy by a nickel plating process, and then modified it with stearic acid. The contact angle of the prepared super-hydrophobic surface is as high as $160.8 \pm 1^{\circ}$, and the rolling angle is 1.8 \pm 1°, showing good long-term stability. Excellent corrosion resistance and self-cleaning function on the surface in 3.5% NaCl solution. Zhang ^[16] et al. Used electrochemical deposition to form super-hydrophobic surfaces in the form of gold clusters on the surface of magnesium alloys. The surface morphology is shown in Figure 2. In electrochemical deposition, polyelectrolyte multilayers were used as pre-made substrates to adjust the morphology of gold clusters. Only the surface covered with dendritic gold clusters showed super-hydrophobicity and the contact angle was greater than 150 °.



Figure 2. SEM photo of dendritic gold clusters on superhydrophobic surface ^[16]

2.4 Oxidation Method

The oxidation method mainly includes micro-arc oxidation and anodization. Anodizing refers to the electrochemical oxidation of metals or alloys. The process of forming an oxide film on the metal (anode) under the corresponding electrolyte and specific process conditions due to the applied current. Micro-arc oxidation is also called plasma electrolytic oxidation. Through the combination of electrolyte and corresponding electrical parameters, on the surface of metals such as aluminum, magnesium, titanium and their alloys, a film layer mainly composed of a base metal oxide is grown in situ by relying on the transient high temperature and high pressure generated by arc discharge. The film has very good adhesion to the base material. Zhang ^[17] et al. Successfully prepared a superhydrophobic coating based on calcium stearate by direct current and pulse electrodeposition on an anodized magnesium oxide alloy, and its static contact angle was about 158°. The corrosion resistance of the coating matrix in simulated body fluids was also investigated. The results show that the coatings prepared under different deposition methods and working cycles show different corrosion resistance. A coating with a 50% duty cycle in pulse mode provides optimal corrosion protection for the substrate. Cui ^[18] et al. Used micro-arc oxidation to treat the surface of magnesium alloys to obtain micron-sized porous structures, and hydrophobically modified them with low surface energy materials to finally obtain super-hydrophobic surfaces with a contact angle of 151.5 °. With the extension of the micro-arc oxidation processing time, the surface structure (see Figure 3) appears to be closed. After 11 days in a 3.5 wt.% NaCl solution, only the edge portions were pitted. The results show that the surface has good long-term corrosion resistance.



Figure 3. Surface morphology and contact angle of different micro-arc oxidation coatings^[18]

2.5 Sol-Gel Method

The sol-gel method is to use a compound containing a highly chemically active component as a precursor, uniformly mix these materials in a liquid phase, and perform a chemical reaction of hydrolysis and condensation. A stable transparent sol system is formed in the solution. The sol slowly polymerizes between the aged colloidal particles to form a gel with a three-dimensional network structure. The gel network is filled with a solvent that loses fluidity and forms a gel. The gel is dried and sintered to prepare molecular and nano-substructure materials. Hu^[19] et al. Successfully prepared tetraethoxysilane and methyltriethoxysilane as precursors by combining micro-arc oxidation (MAO) and sol-gel method on magnesium alloys. A MAO layer having a highly porous surface is formed as an intermediate layer to ensure excellent adhesion of the superhydrophobic silica film to the substrate.

Superhydrophobic surface is mainly composed of micro-nano structure and low surface energy modification. For magnesium alloy superhydrophobic polymer composite coatings, the micro-nano structure is mainly composed of inorganic nanoparticles, and the surface modification is mainly performed by substances with low surface energy. The polymer provides the connection between the nanoparticles and the metal substrate or the underlying coating, so that the coating and the metal substrate have stronger adhesion.

3. Superhydrophobic Polymer Composite

Polymer composite protective coatings are usually multilayer systems consisting of at least a primer and a topcoat ^[20]. The primer provides primary corrosion inhibition and adhesion, and the topcoat is responsible for environmental (such as UV, water, mechanical, microbial, etc.) resistance and different functions as well as some aesthetic requirements ^[20]. Polymer composites with super-hydrophobic function are materials that are macro- or micro-composite together through polymers and micro-nano particles with two or more components with different properties. The two main components are polymer-based coatings and micro-nano particles. Polymer-based coatings are mainly polymers with hydrophobic properties, which can enhance the adhesion between the polymer coating and the substrate, provide primary corrosion protection, and are one of the key components of anticorrosive coatings. The micro-nano structure of micro-nano particles can not only provide the roughness required for superhydrophobic, but also increase the wear resistance and mechanical properties of the coating. The modification of low surface energy substances can be performed by modifying nanoparticles or by modifying the entire composite coating.

3.1 Polymer

3.1.1 Polydimethylsiloxane (PMDS)

Polydimethylsiloxane (PMDS) is a kind of high-molecular organosilicon compound, which has a certain degree of hydrophobicity and is easy to be concentrated at the interface of air. Therefore, in superhydrophobic polymer composites, polydimethylsiloxane (PMDS) is one of the common polymers. Li ^[21] et al., made a strong superhydrophobic coating by spraying a fluorine-free suspension composed of epoxy resin (EP), polydimethylsiloxane (PDMS) and modified SiO2 on a substrate (Figure 4). The coating exhibits excellent superhydrophobicity with a contact angle of 159.5 ° and a sliding angle of 3.8 °. The prepared super-hydrophobic EP-PDMS @ SiO 2 coating exhibits good mechanical durability and remains super-hydrophobic after exposure to harsh conditions.



Figure 4. Schematic diagram of superhydrophobic coating manufacturing process ^[21]

3.1.2 Polylactic Acid (PLA)

Polylactic acid (PLA) is a non-toxic and non-stimulating synthetic polymer material of hydrophobic aliphatic polyesters. It has good transparency, biodegradability, biocompatibility and thermoplasticity. It is a recyclable resource, and is therefore widely used. Because the hydrophobic nature of polylactic acid itself can play an effective antibacterial role in medical applications, using polylactic acid as the main component of superhydrophobic composite materials in biodegradable materials can not only effectively antibacterial, and it also has a certain effect on controlling cell growth or inhibiting cell growth. The resulting superhydrophobic polymer composite coating also has good corrosion resistance. So far, polylactic acid (PLA) has been mainly used in magnesium and magnesium alloy biodegradable materials. Kannan et al. [27] proposed to perform CaP conversion on MgAl alloy AZ91 by electrodeposition, and then impregnate to prepare CaP / PLA superhydrophobic composite coating with contact angle up to 153 $^{\circ}$.

3.1.3 Polytetrafluoroethylene (PTFE)

Polytetrafluoroethylene (PTFE), commonly referred to as "non-stick coating" or "easy-to-clean material". It has the characteristics of acid and alkali resistance and resistance to various organic solvents, and is almost insoluble in all solvents. At the same time, polytetrafluoroethylene has the characteristics of high temperature resistance. It is currently known that no solid material can adhere to the surface. It is also a solid material with the smallest surface energy. Its surface energy friction coefficient is extremely low. Therefore, it has a considerable application potential in polymer composites with superhydrophobic functions.

According to the characteristics of polytetrafluoroethylene itself, Mashtalyar et al. ^[30], through plasma electrolytic oxidation (PEO), followed by a suspension of telomere solution of superdispersed polytetrafluoroethylene (SPTFE) and tetrafluoroethylene (TFE) Surface modification of medium-treated magnesium alloy. The electrochemical, tribological and wetting properties of the obtained composite protective coatings were studied. Compared to unprotected magnesium alloys and base PEO coatings, the manufactured coatings reduce the corrosion current and wear density by orders of magnitude. This versatile coating has high corrosion resistance and a good coefficient of friction under dry abrasion conditions, thereby extending the area of application of magnesium alloys. According to the electrochemical impedance spectroscopy data and calculated parameters of equivalent circuit elements, the SPTFE treatment of PEO coating results in an increase in the thickness of the composite coating.

The protective performance of the composite coating obtained by the SPTFE treatment is four orders of magnitude higher than that of the initial coating formed by the PEO method. The corrosion current density and wear of the composite coating were reduced by three orders of magnitude. In addition, the coating obtained by impregnating PEO and SPTFE telomer dispersion has superhydrophobic properties, the contact angle is greater than 152°, and the wettability of water and other liquids is poor, which further enhances the corrosion resistance of the coating.

The polymer in the protective coating of the polymer composite material with super-hydrophobic function, in addition to having a certain degree of hydrophobicity itself, can also be used for the adhesion between the coating and the substrate.

3.2 Nanoparticles

Nanoparticles refer to particles with a particle size between 1-100nm (nanoparticles are also called ultrafine particles). It belongs to the category of colloidal particle size. They are located in the transition zone between atom clusters and macroscopic objects, between the microscopic system and the macroscopic system, and are a group composed of a small number of atoms or molecules. Therefore, they are neither a typical microscopic system nor a typical macroscopic system. The distinguishing feature of nano particles from the structure of macroscopic objects is that their surface area accounts for a large proportion, and the surface atoms have neither long procedures nor short procedures. It can be considered that the state of the atoms on the surface of the nanoparticle is closer to the gaseous state, and the atoms inside the particle may be in an orderly arrangement. Even so, due to the small particle size and large surface curvature, a high Gilibs pressure is generated inside, which can cause some deformation of the internal structure. This structural feature of nanoparticles makes it widely used in hydrophobic functional protective coatings.

3.2.1 Nano Silica

As an excellent structural and functional material, nano-silica shows excellent surface activity, high specific surface area, high temperature resistance, and non-toxic and pollution-free performance. Due to the high surface energy of silica, it is easy to agglomerate during use, and the nano-silica has poor compatibility with most polymer materials and cannot achieve the desired composite material properties. Surface modification of nano-silica not only effectively prevents agglomeration, but also improves the compatibility and dispersion stability of nano-silica and polymer materials. At the same time, the surface modification can also make the surface of nano-silica have special functional groups, and the binding force with the polymer is enhanced, so that the composite material has more special functions. For example: Wu^[32] et al. fabricated a composite material containing a diamond dodecahedron zeolite imidazolate skeleton (ZIF-8 @ SiO2) on an AZ31 magnesium alloy by chemical etching and dip coating methods to enhance stability and corrosion resistance. (Figure 5) Modify the hydrophobic Cetyltrimethoxysilane (HDTMS) on ZIF-8 @ SiO2 to improve its hydrophobicity. The results show that various liquids can stabilize and maintain contact angles above 150 ° on their surfaces. Hydrophobic modification is achieved in alkoxysilanes. Finally, the etched Mg alloy with a micron morphology was immersed in a hydrophobic ZIF-8 @ SiO2 n-hexane solution. The super-hydrophobic surface with a layered structure made of magnesium alloy not only has good water resistance, but also has excellent corrosion resistance, mechanical properties and chemical stability.



Figure 5. Hydrophobic process and experimental flow on ZIF-8 @ SiO2 ^[32]

In addition, Zhang ^[33] et al. prepared a micro-nano-structured polydimethylsiloxane and silicon dioxide (PDMS / SiO2) composite coating on the surface of a magnesium alloy with two different-sized SiO2 nanoparticles by a simple painting method. Floor. The effects of 40nm and 50-250nm SiO2 ratio on the wettability, wear resistance and corrosion resistance of composite coatings were studied. By using scanning electron microscope (SEM), Fourier transform infrared spectroscopy (FT-IR), contact angle measuring instrument, sandpaper weight device and other tools, the morphology, composition, water contact angle, abrasion resistance and corrosion resistance of the composite coating were measured, and mechanical and thermal stability were also obtained by finger wiping, scratch test and thermogravimetric analysis. The results show that the PDMS / SiO2 composite coatings prepared under optimized parameters are stable and superhydrophobic, and their contact angles can be maintained at a high level after 50 sanding cycles. (138-150 $^{\circ}$ and 133-153 $^{\circ}$). The composite coating shows good resistance to cuts and finger touch. And electrochemical experiments show that a strong coating can significantly improve the corrosion resistance of magnesium alloys.

3.2.2 Nano Zinc Oxide

Nano zinc oxide is a new type of multifunctional inorganic material. In recent years, nano zinc oxide has been widely used in textiles, coatings and other fields, especially in biomedicine. Like nano-silica, nano-zinc oxide can also improve the corrosion resistance and abrasion resistance of protective coatings to achieve the same effect. For example, Zhou^[35] et al. proposed a new idea to imitate the process of weeding (see Figure 6). It solves the problems of low adhesion, weak abrasion resistance and poor corrosion resistance of general super-hydrophobic coatings. The ratio of epoxy resin to ZnO seeds is strictly controlled so that ZnO seeds are not completely coated. On this basis, a clustered ZnO coating composed of cross-index ZnO rods was prepared. After being modified with stearic acid, the coating showed superhydrophobicity, the contact angle was 163°, and the coating showed excellent abrasion resistance and obvious robustness. The coating has excellent corrosion resistance due to the epoxy / ZnO seed and the barrier layer of clustered ZnO. At the same time, the clustered ZnO / epoxy resin coating prepared like the rooting of seeds has good robustness, which can adapt to various harsh environments, such as tape peeling, friction between objects, etc., expanding the application range of superhydrophobic coatings.



Figure 6. Schematic diagram of the preparation process of clustered ZnO coatings ^[35]

3.2.3 Nano Titanium Dioxide

Nano titanium dioxide has self-cleaning, anti-aging,

chemical stability and thermal stability, so it is also widely used in anticorrosion control. Huang [37] et al. used atomic laver deposition (ALD) technology to prepare a laver of TiO2 nano-film with controlled thickness on the surface of AZ31 magnesium alloy (see Figure 7), which can adjust the corrosion behavior of AZ31 magnesium alloy. Compared with the untreated magnesium alloy, the corrosion current density (icorr) of the magnesium alloy covered with TiO2 over 200 cycles can be reduced by 58%, and as the thickness of the nanofilm reaches 63 nm (400 cycles), the corrosion current density further decreases to 74%. Subsequently, the 3-aminopropyltriethoxysilane (APTES) cross-linked conversion layer was modified by the dipping method. A dense silane coating can be formed on the TiO2 nanofilm, which can seal the pinholes of the TiO2 nanofilm, and further adjust the substrate's corrosion barrier behavior. In a TiO2 / silane composite coating, the corrosion current density can be reduced by about two orders of magnitude. Making adjustable corrosion rates a reality can be attributed to the precise control of metal oxide nanofilm thickness and the additional protection of compact silane coatings.



Figure 7. Preparation of superhydrophobic surface modification method ^[37]

In summary, nanoparticles can provide the rough structure required for superhydrophobic surfaces. The ability to attach to the surface of the coating through polymers also provides some abrasion and mechanical durability. In recent years, more and more researchers have added inorganic nanoparticles, such as nano-silica, nano-alumina, etc. to the coating in order to enhance the roughness of the coating and to protect the corrosion with self-healing The coating provides space to store healing agents or inhibitors, and has great prospects in the research of smart materials.

4. Conclusion

This article reviews the preparation methods and structural composition of polymer composite coatings with superhydrophobic function for magnesium alloys. To achieve superhydrophobic effect, one is to increase the roughness, but to reduce the surface energy. Polymers provide the adhesion between the coating and the substrate, and also increase the protection of the substrate to a certain extent. Nanoparticles can not only increase the roughness, but also reduce the surface energy of the coating after hydrophobic modification on the surface. In addition, the coating can also be directly hydrophobically modified. So far, the related research of magnesium alloy anticorrosion in the medical field is very promising, and the corrosion control of magnesium alloy is still a major difficulty in the application of magnesium alloy. At the same time, poor adhesion between the coating and the substrate, super mechanical properties, and poor abrasion resistance are all important issues that need to be resolved, and also indicate the direction of development for metal corrosion.

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REVIEW

Convergence Proving of the Theoretical & True Elongation Inequalities by Derivation and Analogy

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ARTICLE INFO	ABSTRACT
Article history Received: 17 March 2020 Accepted: 26 March 2019 Published Online: 31 March 2020	According to LNE, theoretical & true elongation of tensile, and by adopting the increasing function of formulas with the derivation and analogy methods, the elongation formula of $0<(1+\epsilon)^{1/\epsilon} & 0<\epsilon^{1/\epsilon}<1& four convergences are deduced too when \epsilon > 1 and 0<\epsilon<1. The increasing function of LNE <\epsilon and LN(1+\epsilon)< \epsilon and LN(1+\epsilon)> LNE are deduced if \epsilon>1 and 0<\epsilon<1 in material dynamics. Finally, the conclusions of LNE <\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced if LNE +\epsilon of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon)<\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon are deduced to the set of LNE +\epsilon and LN(2+\epsilon are deduced to the set of LNE +\epsilon$
Keywords:	duced together if $\epsilon > 1$ and $0 < \epsilon < 1$.
0<ε<1	
E > 1	
Analysis	
Derivation and analogy	
Elongation	
Inequality convergence	
Proving	
Theoretical and true elongation	
$LN\varepsilon \leq \varepsilon$	
$LN\varepsilon \leq LN(1+\varepsilon) \leq \varepsilon$	
$0 < (1+\varepsilon)^{1/\varepsilon} < e \text{ and } 0 < \varepsilon^{1/\varepsilon} < 1$	

1. Introduction

Under the condition of density invariance, the convergence of the real and theoretical elongation inequality in material mechanics is proved. When the elongation is greater than $0.15\sim0.2$, there is no comparison between the true elongation and the theoretical elongation. ^[1-3] through the function analysis to judge their relationship of increasing function, so as to determine their comparison, in order to clarify their mathematical

comparison.Because we want to know the true elongation of LN $(1+\varepsilon)$ in the mechanics of materials.Although the phenomenon can be known from the experiment, it has not been proved from the mathematical relationship.Therefore, this paper abstracts the phenomenon and proves it mathematically, and finds that there is a certain relationship between them.That is, the true elongation is greater than the theoretical elongation, so that the experimental research becomes mathematical theory plasticity, which is a big proof of this paper. Prove "1 + 1 =?" like goldbach's

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conjecture. In 1742, he wrote to Euler to talk about it. Its mean is what two prime numbers add? From the point of view of mathematics, finally "1 + 2" were proved by Chen Jingrun in a large chapter. It means that any bigger than two is presented by a prime number and multiplying two prime number. But it has some key points for elongation to prove successful, so from the increasing function and analogy is the key to this paper, prove that LN $(1 + \varepsilon) < \varepsilon$ is the core of this article, the last two induced mathematical inequality can also induce even dozens of inequality. Its proof process is not as long as Chen jingrun wrote so deep. The same is true for shrinkage rates, which can be traced back to Chinese database natural science full text 2020.1 (3).

We use these formulas to understand the internal relation problem, carry on the derivation and the analogy method.We prove that $y = LN(1+\epsilon)$ is an increasing function.Critical problems such as $LN\epsilon < LN(1+\epsilon) < \epsilon$ when >0 are proved.

2. The Proof of $0 < (1+\epsilon)^{1/\epsilon} < e \& 0 < \epsilon^{1/\epsilon} < 1$

2.1 The Proof of $y = LN\varepsilon$ being Increasing Function

The discussion situation here is $\epsilon \geq 1$.

If
$$\varepsilon > 1$$
 hasLN $\varepsilon > 0$ (1)

Supposes y= LNx=LNɛ

has
$$\frac{dy}{dx} = \frac{d(LN\varepsilon)}{d\varepsilon}$$
 (2)

since $y = \frac{d(LN\varepsilon)}{d\varepsilon} = \frac{1}{\varepsilon}$

Due to
$$\frac{d(LN\varepsilon)}{d\varepsilon} > 0$$
 (3)

& $0 < 1/\epsilon < 1$ (4)

y= LNx is nereasing function. Its maximum is 1.

2.2 The Proof of $Y = LN(1 + \epsilon)$ being Increasing Function

If $\varepsilon > 1$ (5)

 $LN\epsilon > 0$ (6)

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Has too
$$LN(1+\epsilon) > LN\epsilon$$
 (7)

supposes y = LN(1+x)

so
$$\frac{dy}{dx} = \frac{d[LN(1+\varepsilon)]}{d\varepsilon} = \frac{1}{1+\varepsilon}$$
 (8)

since
$$\frac{dy}{dx} > 0$$
 (9)

So that y=LN(1+x) is increasing function.

2.3 Proves $LN(1+\varepsilon) < LN\varepsilon$

since
$$LN(1+\epsilon)/(1+\epsilon) < LN (1+\epsilon)/\epsilon$$
 (10)

If
$$\epsilon > 1$$
 according to $\epsilon t = LN(1+\epsilon) > LN\epsilon$ (11)

has LN
$$(1+\varepsilon)$$
 /LN ε >LN ε /LN ε =1. (12)

because LN $(1{+}\epsilon)\,$ and LN ϵ is increasing function. From above it has

from (11) it has

$$LN (1+\varepsilon) / LN\varepsilon > 1$$
 (13)

So that
$$LN(1+\varepsilon) > LN\varepsilon$$
 (14)

That is the first result in this paper.

2.4 Proves $LN(1+\varepsilon) < \varepsilon$

2.4.1 When E>1

From $1/\epsilon < 1$ (15)

According to the above increasing function it has

$$LN(1+\varepsilon)/\varepsilon < \varepsilon/LN\varepsilon$$
 (16)

This is the comparison among elongation and true stress when elongation $\epsilon \ge 1$, the above inequality(15) is gained upon inequalities comparison. The relationship may be seen in Figure 1, the biggest difference among LN ϵ & LN (1+ ϵ) is 25.

$$\varepsilon/LN\varepsilon < \varepsilon/\varepsilon = 1$$
 (17)

According to equations (15) & (16) it has

$$1/\epsilon LN(1+\epsilon) < 1$$
 (18)

ie.
$$LN(1+\varepsilon) \le \varepsilon$$
 (19)

This is the important inequality result in this paper among true tress and engineering one when $\varepsilon > 1$.

2.4.2 When 0<ε≤1

Since $\varepsilon t = LN(1+\varepsilon) > LN\varepsilon$ (23)

has LN (1+ ϵ) /LN ϵ >LN ϵ /LN ϵ =1 (24)

Has too LN $(1+\varepsilon)$ >LN ε (25)

If 0<E<1because1/e>1

in equation(12) it is known that $1/\epsilon > \epsilon$ (26)

This is the relationship between true and engineering elongation when $\epsilon > 1$. The above inequality (26) and below (27) is obtained from this search for plasticity to compare which result the inequality (31). the relationships is in Figure 2, the biggest difference is 25.

According to equation, it has been known that when $0 \le 1$ it has LN $\epsilon \le 0$ ie.1/ $\epsilon \ge 1$,

hence $1/\epsilon LN\epsilon < 1$ (27)

has $LN\epsilon \leq \epsilon$ (28)

According to $1/\epsilon LN(1+\epsilon) \le LN2 \le 1$ (29)

and $1/\epsilon > \epsilon$ since $1/\epsilon > 1 > \epsilon$, $LN(1+\epsilon) > 0$

 $0 = LN1 < 1/\varepsilon LN(1+\varepsilon) < 1/\varepsilon LN2 < 1$ (30)

 $LN(1+\epsilon)>1/\epsilon$

Hence $LN(1+\varepsilon) \le \varepsilon$ (31)

It is a important result deduced in this paper when $0 \le \epsilon \le 1$.

From equation (25) &LN ϵ < 0 it has been known

$$LN(1+\varepsilon)/\varepsilon <-LN(1+\varepsilon)/LN\varepsilon <\varepsilon/-LN\varepsilon <\varepsilon/\varepsilon=1$$
(31)

 $-LN(1+\varepsilon)/LN\varepsilon < \varepsilon/\varepsilon = 1$ (32)

Hence according to (25) it has always

 $LN(1+\varepsilon) > LN\varepsilon$ (33)

This is the results between LN ϵ and LN(1+ $\epsilon)~$ mathematics when 0< ϵ ${\leq}1.$



Elongation %

Figure 1. The relations of function of \mathcal{E} , LNE & LN(1+ \mathcal{E}) if $\mathcal{E}>1$



Elongation %

Figure 2. The relations of function of ratio with \mathcal{E} , LNE & LN(1+ \mathcal{E}) If $\mathcal{E}>1$



Figure 3. The relations of function of difference with E, LNE & LN(1+E) If E>1



Figure 4. The relations of function of deviation with \mathcal{E} , LNE & LN(1+ \mathcal{E}) If $\mathcal{E}>1$

2.5 Prove $0 < (1+\varepsilon)^{1/\varepsilon} < e$

Since $LN(1+\epsilon)/|LN\epsilon| > \epsilon/|LN\epsilon|$ (34)

From equation(13) it has

 $1/|LN\epsilon|>1/\epsilon \tag{35}$

hence $|LN\epsilon| > \epsilon$ (36)

Ie
$$LN(1+\varepsilon) \leq LN\varepsilon$$
 (37)

It has that $(1+\epsilon)^{1/\epsilon} < e$, when $1 > \epsilon > 0$ it has $LN\epsilon < 0$; when $\epsilon > 1$ it has $LN\epsilon > 0$. This is discussed above. when $0 < \epsilon \le 1$ it has in (12) and (13)

$$(1+\varepsilon)^{1/\varepsilon} < 1 \tag{38}$$

From equation(10) it has known that

 $\varepsilon^{1/\varepsilon} < (1+\varepsilon)^{1/\varepsilon} < 1 \tag{39}$

When $0 \le \varepsilon \le 1$ it has known that $y' = \varepsilon^x LN \varepsilon \ge 0$, so that $y = \varepsilon^x$ is increasing function.hence

 $0 \le \varepsilon^x$ (40)

Hence it is been proven as below

$$0 \le \epsilon^{1/\epsilon} \le 1$$
 (41)

From (12) it has know that
$$LN(1+\epsilon)^{1/\epsilon} < 1=LNe$$
 (42) i.e.

$$(1+\varepsilon)^{1/\varepsilon} < e$$
 (43)

In terms of the above proven $LN(1+\epsilon)$ being increasing function it has

 $LN(1+\varepsilon)/\varepsilon > LN1/\varepsilon = 0 \tag{44}$

Due to $LN1/\epsilon=0$ (45)

It has $0 < (1+\varepsilon)^{1/\varepsilon} < e$ (46)

This is a mathematical inequality in this paper, it expresses that below relationship $(1+\varepsilon)^{1/\varepsilon} \le 0 \le (1+\varepsilon)^{1/\varepsilon}$.

It expresses that $(1+\epsilon)^{1/\epsilon}$ mathematical meaning in the scope of 0 & e=2.7.

 $0 < 1/(1 + \varepsilon) \tag{47}$

So that $LN(1+\varepsilon) \le \varepsilon$ (48)

i.e.

$$LN(1+\varepsilon)^{1/\varepsilon} < 1 = LNe$$
(49)

i.e.

 $LN(1+\varepsilon)/\varepsilon > LN1/\varepsilon = 0$ (51)

So
$$0 < (1+\varepsilon)^{1/\varepsilon} < e$$
 (52)

2.6 Prove $0 < \varepsilon^{1/\varepsilon} < 1$

It is known above that $(1+\varepsilon)^{1/\varepsilon} < e$, if $1 > \varepsilon > 0$, LN $\varepsilon < 0$; If $\varepsilon > 1$, LN $\varepsilon > 0$. from (12) and (10) it has

$$(1+\varepsilon)^{1/\varepsilon} < 1 \tag{53}$$

From (10) & (12) it has

$$\varepsilon^{1/\varepsilon} < (1+\varepsilon)^{1/\varepsilon} < 1 \tag{54}$$

If $\epsilon \!\!\geq 1 \ dy/dx \!\!= \epsilon^x LN\epsilon \!\!> 0,$ so $y \!\!= \epsilon^x$ is increasing function. hence

 $0 \le \epsilon^{x}$ ----- (55)

It proves the relationship of $0 \le \epsilon^{1/\epsilon} \le 1$ (56)

3. Discussion

Figure 1~4 is the comparison of the true elongation and the elongation when the is not less than 1. The inequality (13) above and the inequality (14) below are the plastic relations obtained by comparing the inequality (17). Their sizes are shown in figure 1, and the comparison between the two is shown in figure 2. The maximum ratio between LN ϵ and LN(1+ ϵ) is over 25. According to figure 1, LN(1 + ϵ) is greater than LN ϵ and the convergence trend is 700%.

The biggest difference is shown in Figure 3 for LN (1 + \mathcal{E}) and \mathcal{E} is 4 for LN \mathcal{E} and LN (1 + \mathcal{E}) is 2.As shown in figure 4 the maximum deviation for LN (1 + \mathcal{E}) and \mathcal{E} is 7 for LN \mathcal{E} and LN (1 + \mathcal{E}) is 1.Therefore, if the elongation is greater than 1, the value drops rapidly.When the elongation is greater than 130%, the trend is flat.

Figure 5~8 shows the comparison of the true elongation and the elongation when the is less than 1. The inequality (13) above and the inequality (14) below are the plastic relations obtained by comparing the inequality (17). Their sizes are shown in figure 5, and the comparison between the two is shown in figure 6. The maximum ratio for LN ϵ and LN(ϵ) is 25. According to Figure 5, LN(1+ ϵ) is greater than LN ϵ .

The biggest difference is shown in Figure 7 for LN (1

+ \mathcal{E}) was 0.5 for LN(1+ \mathcal{E}) and \mathcal{E} & and LN (1 + \mathcal{E}) is 2.5.As shown in figure 8 the maximum deviation for LN (1 + \mathcal{E}) and \mathcal{E} is 7 for LN \mathcal{E} and LN (1 + \mathcal{E}) is 2.So if the elongation is less than 1, the value drops sharply. When the elongation is less than 70%, the trend is flat.

Because dy/dx= $1/\epsilon x^{1/\epsilon \cdot 1} > 0$, when $\epsilon > 1$ y= $x^{1/\epsilon}$ is increasing function. at the same time to prove the inequation (56).



Figure 5. The relations of function of E, LNE & LN(1+E) if $\varepsilon < 1$



Figure 6. The relations of function of ratio with \mathcal{E} , LNE & LN(1+ \mathcal{E}) if $\mathcal{E} < 1$



Figure 7. The relations of function of difference with &&LN&, LN&&LN(1+&) if $\&\le 1$



Figure 8. The relations of function of deviation with E& LNE, LNE&LN(1+E) if ε< 1

4. Conclusions

1. The inequality between true and theoretical elongation in material mechanics is proved. The inequality between LN(1+ ϵ)> LN ϵ and LN(1+ ϵ)< ϵ is proved when 0<e.

2. The convergence of $0 < (1+\epsilon)^{1/\epsilon} < \epsilon \& 0 < \epsilon^{1/\epsilon} < 1$ is derived from $y=\epsilon^x \& y=x^{1/\epsilon}$ is an increasing function. In mechanics of materials prove LN $(1+\epsilon) < LN \epsilon$, LN $(1+\epsilon) < \epsilon$ and LN $(1+\epsilon)/(1+\epsilon) < LN (1+\epsilon)/\epsilon < \epsilon/\epsilon$ is established.

3. the greatest difference in LN(1+ \mathcal{E}) and \mathcal{E} is 4 while LN \mathcal{E} and LN(1+ \mathcal{E}) is 2. The greatest deviation in LN(1+ \mathcal{E}) and \mathcal{E} is 7 while LN \mathcal{E} and LN(1+ \mathcal{E}) is 1. The maximum ratio between LN \mathcal{E} and LN(1+ \mathcal{E}) is more than 25.

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ARTICLE Effect of Anisotropy, Temperature, Strain Rate on Deep Drawing Using Conical Die

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ARTICLE INFO ABSTRACT

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1. Introduction

onical type die raises the limiting drawing ratio of deep drawing cups. It can manufacture various cross sections at die cylindrical throat to permit best metal flow from the conical section to desired shape at the die end.

Mihael Volk et al. ^[1] presented few aspects of blank-holder pressure patterns in conventional deep drawing based on finite element assessment. In all models, ABAQUS software was used with full 3-D model to investigate anisotropy and wrinkling of the parts. The blank is made of aluminum 5182 alloy, this is an elastic-plastic analysis. Friction based on Coulomb friction model with 0.1 coefficients between the blank and the various tool. Lihui Lang et al. ^[2] observed influences of many parameters that start wrinkling on a cylindrical cup. The goal of this study is to show the influence of

This paper covers the role of anisotropy, temperature, and strain rate on the flow behavior of the material when a conical die is used instead of conventional blank holder. The effect of anisotropy was investigated using Lankford's coefficient (r) in three directions (0°, 45°, and 90°). The effect of working temperatures (Room temperature, 100°C - 300°C) on drawing stress and strain rate sensitivity on punch pressure were also investigated in detail. ANSYS APDL was used to investigate the effects of temperature, strain rate and anisotropy. The simulation results have confirmed that the strain variation in the direction of r_0 and r_{45} are more than the variation of r_{90} .

different factors such as BHF, punch and die radius, and friction on the wrinkling of cylindrical parts. Marisa P. Henriques et al.^[3] describes the initiation of wrinkles during sheet metal forming. ABAQUS software was used for FEA. The results showed that, correct prediction of wrinkling defects.

In this present study, effects of strain rate, anisotropy and temperature on plastic deformation characteristics of AISI 304 grade steel blanks analyze using ANSYS APDL simulation software.

2. Methodology of Present Work

In this present study, the methodology, consists of a number of steps and sub-steps (Figure1). Starting from the creation of a 3-Dimensional axisymmetric model and then defining input parameters such as material properties, parameters, boundary conditions, meshing, and iterations.

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Figure 1. Flow chart of methodology

3. Creation of Tool and Work Piece Geometry

In this investigation, a finite element model is created using ANSYS APDL pre-processor. The axisymmetric 3-Dimensional model developed for analysis is shown in the below figure 2.



Figure 2. FE model for conical die without blank holder

4. Investigation of Anisotropy Problems in Deep Drawing using FEM

This section discusses the results of the numerical simulations by considering plastic anisotropy of stainless steel sheets. A 3-D parametric FE model was built using the ANSYS APDL. The material used in this work was sampled from a rolled sheet of 1.5 mm thickness AISI 304 stainless steel.



Figure 3. Experimental and simulated cylindrical cup

In general, the anisotropic behavior of sheet metals is evaluated using Lankford's coefficient, (r) commonly known as plastic anisotropy parameter ^[4-6]. A uniaxial tensile test is used to determine the r-value by using two extensometers, one in the longitudinal direction and the other one in the transverse direction. The ratio between two major strains are determined using constancy of volume i.e $\varepsilon_l + \varepsilon_w + \varepsilon_t = 0$ and expressed as below –

$$r = \frac{\mathcal{E}_{w}}{\mathcal{E}_{t}} \tag{1}$$

Where, r is Lankford's plastic anisotropy ratio, ϵ_w and ϵ_t are the strain values in width and the thickness directions respectively. The true strain values are also be expressed as –

$$\varepsilon_l = \ln\left(\frac{l_0}{l_f}\right) \tag{2}$$

and

$$\varepsilon_w = \ln\left(\frac{w_0}{w_f}\right) \tag{3}$$

Where Lo is the original length of the tensile specimen, Lf is the final length of the specimen; W_o and W_f are the initial and final width of the tensile specimens respectively. Thus, the equation (4) may be rewritten as –

$$r = \frac{\ln\left(\frac{w_0}{w_f}\right)}{\ln\left(\frac{l_f w_f}{l_0.w_0}\right)}$$
(4)

Often, the r-values are determined at different directions of the sheet metal i.e. at 0, 45 and 90 degrees from the rolling direction, as shown in the inset of Figure4.The average value is called normal anisotropy, which is expressed as (\bar{r}) -

$$\overline{r} = \frac{\left(r_0 + 2r_{45} + r_{90}\right)}{4} \tag{5}$$

The experimental results of tensile specimen obtained from a stainless steel sheet at different directions are shown in Figure4 and values of Lankford's coefficient, strain hardening exponent and other mechanical properties are presented in table 1.



Figure 4. True stress-strain curve for AISI 304 Stainless steel at r_0 , r_{45} and r_{90} degrees from the rolling direction

Orientation	Ultimate tensile strength (in MPa)	Hardening expo- nent (n)	Lankford's coeffi- cient (r)
0°	528	0.43	0.76
45°	524	0.41	1.38
90°	530	0.43	0.83

Table 1. Plastic Anisotropy parameter of AISI 304

As determined through the experimental measurements the AISI 304 sheet metals shows higher normal anisotropy (\bar{r}). This suggests that the cup wall of this material can withstand higher load without excessive thinning and fracturing ^[7-10]. The values of planar isotropy (Δr), suggests that ear formation will occur ± 45°.

5. Simulation Results by Considering Plastic Anisotropy

The variation in the deep drawing behavior of AISI 304 sheet metal with respect to plastic anisotropy is investigated through simulation studies. The possibilities of wall thinning, fracturing and earring were investigated by considering plastic anisotropy i.e. by using Lankford coefficients.



Figure 5. Effect of plastic anisotropy on thickness strain of cylindrical cup simulated using FEM

A cylindrical cup drawn using a conical die instead of a conventional blank holder is considered for simulations studies. The variation in the thickness strain at different regions of the cup drawn by considering plastic anisotropy is shown in Figure 5.

6. Effect of Temperature on Deep Drawing of Cylindrical Cup Using Conical Die

Warm working is the plastic deformation of metal at temperatures below the temperature range for recrystallization and above the room temperature. In this investigation, 1.5 mm thick circular blank of stainless steel AISI 304 were warm deep drawn and the influence of temperature on the deformation behavior of material and the drawing stress which is required to draw the component was studied by FEM software ANSYS APDL.

Influences of temperature on drawing stress

Temperatures ranging from 25°C (room temperature) to 300°C in various temperatures of 100°C, 200°C, and 300°C were applied during forming and the effect on the required drawing stress was studied. It is observed from figure 6 that increase in temperature decreases the drawing stress approximately by 33%. Figure 6 is showing that, stress is continuously decreased with increasing temperature.



Figure 6. Effect of temperature on drawing stress

Figure 7 (a-c) is shows effects of temperature on principal stress on cylindrical cup. Result shows that the principal stress is observed to decrease with increasing temperatures, this is because the flow stress is better on high temperatures. Figure 7 (a) shows at room temperature the principal stress is 60 MPa but after increment of temperature the stress decreases upto 54 MPa. This shows that the temperature affect the formation of deep drawing process.



Figure 7. Effect of temperature on stress (a) Room temperature (b) 100° temperature (c) 200° temperature

Figure 7 is shows effects of temperature on principal stress on cylindrical cup. Result shows that the principal stress is observed to decrease with increasing temperatures, this is because the flow stress is better on high temperatures. Figure 7 (a) shows at room temperature the principal stress is 60 MPa but after increment of temperature the stress decreases upto 54 MPa. This shows that the temperature affect the formation of deep drawing process.

7. Effect of Strain Rate on Deep Drawing Using Conical Die

The strain rates can be determined if the velocities are known. The stresses and strains are computed by using the strain rates. For stainless steel metal, a better description of the strain-rate sensitivity of flow stress is given by–

$$\sigma_f = K \cdot \varepsilon^n \cdot \left(1 + \frac{\varepsilon_1}{\varepsilon_2}\right)^m \tag{6}$$

Where, σ_f is flow stress (in MPa), K is strength coefficient (in MPa), ϵ is the flow strain, ϵ_1/ϵ_2 is the ratio of strain rate and m is the strain rate sensitivity.



Figure 8. Two possible effects of strain rate sensitivity on stress–strain curves

Figure 8 shows the strain rate sensitivity. The tensile test was conducted for different strain rates (high as well as low strain rates). Both the values of stress-strain used as input data in FE simulation.

8. Conclusions

The significant credentials of the present study are listed below:

The values of planar isotropy (Δr), suggests that ear formation will occur $\pm 45^{\circ}$. The maximum thickness strain is observed at to the cup wall region in comparison to the cup bottom. The simulation results have demonstrated that the strain variation in the direction of r_0 and r_{45} are more than the variation of r_{90} , an increase in temperature decreases the drawing stress approximately by 33% but temperature does not affect the formation of tearing in the thinning section, and at low strain-rate punch pressure decreases and punch pressure rise in high strain rate.

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