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Enhancing Performance of Improved Wear and Corrosion Resistance in Nickel-Based Super Alloys via Zirconium Coating

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***Abstract*:**

An essential industrial procedure used to protect base materials from wear, corrosion, and numerous other surface-related degradation phenomena is surface modification via thin film deposition. For their hardness and resistance to corrosion, thin hard coatings like zirconium (Zn) coatings have been utilized to make tool dies. Super alloys based on nickel are provided in a heat-treated state, often hardened and tempered to meet the needs of a certain application. Precision items called tool dies have final shapes and dimensions that must be accurate to within a few microns in order to produce parts. The chemical composition affects the machinability of the nickel-based super alloys in distinct ways. This research paper aims to cover nickel-based super alloy components with zirconium. It is crucial to demonstrate how various sputtering circumstances contribute to the necessary microstructural characteristics. Sputtering parameters efficiently control the thin film's microstructural properties. The current work attempts to optimise the zirconium thin film coating on a nickel-based super alloy by examining the influence of process parameters on coated surface attributes. The Pin on Disc and Salt Spray Test as well as the Vikers Hardness Tester will be used to evaluate the coating's properties.

Key words:*PVD technique; Sputtering;Deposition Improvement; Coating; Zirconium*

1.Introduction

When two surfaces come into contact, wear occurs on both surfaces. Surface engineering is a cost-effective way for producing materials, tools, and machine parts with the necessary surface qualities, such as wear and corrosion resistance. Consumers and industry often concentrate on the worn surface that has the biggest influence on their personal financial status and view the other surface as abrasive. Plasma-assisted coatings have proven to be very effective for dependable, cost-effective surface restructuring procedures. The two main types of thin film deposition processes are Chemical Vapour Deposition (CVD) and Physical Vapour Deposition (PVD). The films in this work were created using PVD processes.

PVD refers to a wide variety of deposition methods in which the initial material is in the solid phase and is subsequently changed to the vapour phase. Nearly all techniques require a low pressure (vacuum) in order to prevent imperfections in the film as well as the loss of energy of particles released from the solid source owing to collisions. This is done mostly to keep the blasting ions or particles in evaporation or sputtering at a sufficient energy level. PVD processing is done at temperatures between 150 and 500 °C in a high vacuum. Metals like titanium, chromium, and aluminium that are high-purity solid coating materials are vaporised by heat or by being bombarded with ions (sputtering). In addition, a reactive gas is supplied, such as nitrogen or a gas containing carbon, which reacts with the metal vapour to generate a compound and deposit a thin, highly adherent coating on the tools or components. The components are rotated about numerous axes at a constant speed in order to achieve a consistent coating thickness.

Zr-702 alloy was coated with plasma electrolytic oxide at two distinct process times, and the coatings' corrosion and wear behaviors were then assessed. According to test results, coating Zr-702 significantly increased its corrosion and wear resistance. It has been found that coatings grown over longer periods of time had higher load-carrying capacities and so offered better wear resistance. On the other hand, a shorter PEO process time resulted in fewer coating structural flaws and improved corrosion resistance.[1] On a Si (100) wafer and an XC100 steel substrate, Cr-Zr-N films were created utilising an R.F. reactive magnetron sputtering technique without the use of heat.The findings demonstrate that the coexistence of (Cr-N, Zr-N) crystallographic orientation mixture affected the film structure as Zr content increased. The films created a solid solution of (Cr, Zr) N in which Zr atoms replaced Cr atoms.[2] Due to its advantages, which include excellent chemical stability (including resistance to oxidation and hydrothermal corrosion), low thermal neutrons absorption cross-section, and excellent adherent, Cr coating research is unquestionably moving forward the fastest in the world. We investigate the oxidation, diffusion, and mechanical properties of Cr-coated Zr alloys in nuclear reactors under both normal operating conditions and accident scenarios.[3]

Sputtering and evaporation are the two basic methods used in PVD to extract the particles from the target, respectively. When compared to the sputtering process, evaporation typically has lower atomic energy, a higher vacuum pressure requirement, fewer gases adsorbed into the coatings, a much more directional nature, particle transfer with a higher mass (larger grains), more oriented grains, a lower adhesion to the substrate, and higher deposition rates. Because of this, thick films and industrial applications are typically better suited for the evaporation process than those where the surface morphology is crucial to quality. Additionally, contaminants may be found in the crucible and may be transferred there from the items to be coated, reducing the purity of the coatings produced.[4] In PVD processes, the target material is broken down into atomic particles through a thermal physical collision process and directed to the substrates in a vacuum environment or gaseous plasma at low

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pressure, where they condense to form a physical coating.[5]With a wide variety of applications already fully established, PVD is a commonly employed method for the deposition of thin films regarding many demands, including the improvement of tribologicalbehaviour, optical enhancement, appearance upgrading, and many other disciplines.[6]

The industry is becoming anxious about how much energy the procedure uses. The PVD technique uses more energy than the CVD procedure, according to studies on energy use and material flows in hard coating deposition processes. However, there are techniques to cut back on energy use, like reusing target materials or recovering leftover heat via heat exchangers. Cathode consumption accounts for more than half of the total energy needed in the deposition cycle in the case of the Sputtering Magnetron deposition process, where the coating phase alone can use nearly three-quarters of the total energy. Heating, etching, and refrigeration therefore make relatively less contributions.[7] A study on electrochemical corrosion testing was carried by the researchers concentrating on galvanised steel sheets with a thin top coating of amorphous zirconia produced by spray pyrolysis. The study looked at how well these coated sheets corroded in both saline and acidic environments. The findings show that the zirconia layers applied to aluminized steel sheets by spray pyrolysis efficiently served as a defence against corrosion in acidic conditions. However, it was shown that in saline environments, their protective impact was restricted. As a result, it may be said that zirconia coatings offer clear corrosion protection in acidic settings but lose some of their efficiency in alkaline environments. [8]

The study examined the chemical, physical, and mechanical characteristics of nickel-based super alloys. It specifically focused on reviewing the mechanical properties and deformation mechanisms associated with these alloys, such as tensile properties, creep behavior, fatigue resistance, and cyclic crack growth.[9] The authors' conclusion was that the addition of Zr to Pt-modified aluminide coatings resulted in improved resistance to spallation of the Thermally Grown Oxide (TGO) layer caused by Zr-rich oxide pegs. As a result, the sample exhibited superior resistance to cyclic oxidation compared to the Pt-modified aluminide coating without Zr addition. The study specifically investigated the effect of Zr addition to Pt-modified aluminide coatings. [10] In a study examining the wear behavior of nickel, iron, and cobalt-based superalloys using a pin-on-disc wear test, it was observed that nickel-based superalloys exhibited higher wear rates compared to cobalt and iron-based superalloys. This higher wear rate was attributed to the formation of NiO during the wear process.[11]

In molten fluoride salts, the corrosion behaviour of different superalloys based on nickel was investigated. Based on the examination, it can be said that Cr was the main element that dealloyed in Ni-based alloys when they were exposed to molten fluoride salts, and that Mo improved the alloys' ability to resist corrosion. The study also emphasised the significance of properly purifying the salts and graphite crucibles used in the corrosion process as well as the presence of air during the corrosion process, both of which might affect the corrosion outcomes.[12]Galvanostatic treatment of the 7075 alumina alloy led to the creation of alumina-zirconia coatings. The study's findings showed that the length of the plasma electrolytic oxidation process had a significant impact on the properties of the coatings.[13] The influence of various types of microstructures and metallic coatings on the surface was investigated, and as a result, it was concluded that Thermal Barrier Coatings (TBC) with either a nanostructure or a conventional microstructure showed the most promising performance in terms of insulation properties within the combustion chamber.[14]

The wear behavior of nickel-based superalloys with different compositions and under various conditions was examined. The study revealed that the wear of nickel-based superalloys is influenced by temperature, indicating temperature-dependent wear characteristics. Additionally, it was observed that as sliding time increases, the coefficient of friction also increases for the Ni-based super alloy coatings, suggesting a correlation between sliding time and friction behavior in these coatings.[15]Based on the literature survey, it has been found that there is limited research available on zirconium coating applied to nickel-based super alloys. This indicates a significant opportunity for conducting further research in this area. Consequently, the objective of this project is to investigate the wear behavior of zirconium-coated nickel-based super alloys. By studying the wear properties of these materials, valuable insights can be gained, leading to potential advancements and improvements in zirconium coating technology for nickel-based super alloys.

2. Selection of Materials

2.1. Nickel Based Super Alloy

Super alloys made of nickel are high-temperature alloys with superior corrosion resistance that are created with service temperatures of 500°C in mind. These alloys often contain sizeable quantities—often up to 10—of various alloying elements. The excellent resistance of super alloys to creep, sulfidation, and oxidation, even when subjected to temperatures near their melting points, is one of their amazing properties. Because of this, they are ideally suited for demanding applications that need outstanding performance in extremely hot and corrosive environments.

Nickel-based super alloys are widely used in demanding applications due to their exceptional properties, including high fatigue strength, thermal stability, and corrosion resistance. Here are some key properties of Ni-based super alloys:

(i) Tensile Strength: Ni-based superalloys exhibit remarkable tensile strength, typically around 690 MPa, which allows them to withstand high mechanical stresses.
(ii) Yield Strength: These alloys also have a high yield strength of approximately 275 MPa, indicating their ability to resist plastic deformation under load.
(iii) High-Temperature Resistance: One of the significant advantages of Ni-based superalloys is their resistance to high temperatures. Unlike many other materials, they maintain their mechanical strength even when exposed to extreme heat, thanks to their inherent thermal protection.
(iv) Oxidation Resistance: When exposed to oxidizing environments, such as high-temperature air, Ni-based superalloys form a protective oxide layer on their surface. This

oxide layer acts as a barrier, preventing further corrosion and enabling the alloys to withstand oxidative attack.

(v) Weldability: Nickel-based superalloys exhibit excellent weldability, making them easier to join compared to other metals and alloys. This characteristic is advantageous in various manufacturing processes and repair operations.

(vi) High-Temperature Strength: Nickel-based superalloys retain their strength at elevated temperatures, withstanding temperatures nearing 1,800 °F (980 °C). This property is crucial for applications that involve prolonged exposure to extreme heat conditions.

Superalloys based on nickel are used in numerous sectors. They are perfect for engine parts and accessories in high-speed, high-friction applications, such as aircraft engines, thanks to their exceptional heat resistance. They are also widely used in steam turbines and combustion chambers because they can resist high temperatures. Due to their resistance to corrosion and material strength, these alloys are used in the nuclear industry for reactor cores and control rods. Due to their capacity to tolerate corrosive conditions and high temperatures, nickel-based superalloys are also used in gas turbines, aircraft components, chemical processing equipment, and the petrochemical industry. Superalloys based on nickel are indispensable for critical applications requiring better performance under demanding circumstances due to their special mix of characteristics.

2.2. Zirconium DiOxide (ZrO_2)

Zirconium dioxide (ZrO_2), also known as zirconia, is a white crystalline oxide of zirconium. It occurs naturally in a monoclinic crystalline structure and is a white powder with high thermal expansion and thermal insulation properties.

ZrO_2 is a crucial zirconium compound widely used in various products due to its exceptional properties. In its natural state, ZrO_2 exists as baddeleyite, featuring a monoclinic crystal structure. Zirconium dioxide is non-magnetic and exhibits high resistance against acids, alkaline solutions, and external influences (chemical, thermal, and mechanical).

Some key properties of ZrO2 include:

(i) High Thermal Expansion: It has a high thermal expansion coefficient (α=11 x 10^{-6}/K), similar to certain types of steel.

(ii) Excellent Thermal Insulation: It possesses excellent thermal insulation properties, characterized by low thermal conductivity ranging from 2.5 to 3 W/mK.

(iii) High Resistance to Crack Propagation: It demonstrates exceptional resistance to crack propagation and high fracture toughness, with values typically ranging from 6.5 to 8 MPa

(iv) Oxygen Ion Conduction: It has the ability to conduct oxygen ions, which is utilized in applications such as measuring oxygen partial pressures in lambda probes.

These remarkable properties make zirconium dioxide highly valuable for a wide range of applications across industries.

3. Experimental Procedure

3.1. Preparation of Test Specimens

Wear tests are essential for verifying the quality, dependability, and lifetime of a product. Manufacturers can find flaws in their products, improve designs, and take well-informed decisions to improve the performance and durability of their products by putting them through simulated wear situations. [16] Hardness testing can be used to determine a material's strength, resistance to deformation, wear, and scratching, as well as its appropriateness for specific applications. It helps with material choice, quality control, process enhancement, and the identification of potential faults or anomalies in the materials.[17]

Water jet cutting is a process that uses a high-pressure jet of water mixed with abrasive particles to cut through materials. The process involves directing the water jet onto the material's surface, which erodes the material and creates the desired shape or size.The cutting parameters for water jet cutting, such as water pressure, abrasive flow rate, and cutting speed, need to be optimized to achieve precise and accurate cuts. The specific parameters depend on the material being cut and the desired quality of the cut. [18] Before conducting the wear and hardness tests, it is important to prepare the cut surfaces of the material. This may involve cleaning the surfaces to remove any debris, contaminants, or residual abrasive particles from the water jet cutting process.

The nickel alloy specimen must be 10 X 10mm in size for the wear and hardness test, as illustrated in the Fig.3.1

Fig.3.1 Wear and Hardness test samples

A corrosion test is used to determine a material's susceptibility to corrosion, which is the progressive degradation or damage brought on by chemical interactions with its surroundings. In order to assess the resilience and performance of materials in varied corrosive environments, corrosion testing is essential in sectors like manufacturing, construction, automotive, and aerospace.For the corrosion test, the nickel alloy specimen must be 40 X 40mm in size, as shown in Fig.3.2

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Fig.3.2 Sample for Corrosion Test

3.2. Thermal Sprayed Coatings

Thermal spraying techniques have been widely applied in a range of engineering fields to preserve and restore components. Recent advancements in equipment and methods have enhanced the quality of thermally sprayed coatings and broadened their scope of applications. The well-known method of thermal spraying is used to apply coatings that are resistant to wear and corrosion in key industry sectors like aerospace, automotive, power generation, petrochemical, and offshore.[19]

The technical dependability of thermal spraying processes has recently been reinforced by improvements in equipment and material quality, which has contributed to a large increase in new applications like biomedical, dielectric, and electronic coatings. As a result, there are many alternatives available to spray coating suppliers in terms of choosing coating materials, thermal spraying equipment, and gas. However, the unique environmental conditions to which the coating will be exposed frequently affect these decisions. Fig. 3.3 and 3.4 shows the general schematic diagram of thermal spray coating processes and experimental set-up for thermal spray coating. Similarly Fig.3.5 and Fig.3.6 depicts the zirconium coated nickel alloy specimens for wear, hardness and for corrosion test.

Fig.3.3 Schematic diagram of Thermal Coating   Fig.3.4 Thermal Spray Coating Set-up

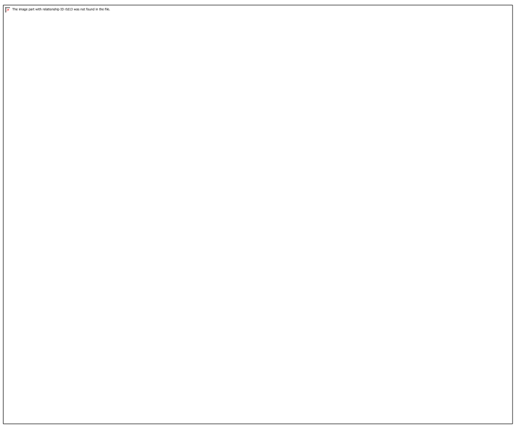

Fig.3.5Zr Coated Wear & Hardness samplesFig.3.6Zr Coated Corrosion Test Sample

3.3. Wear Test

A tool used to test wear is a tribometer, wear tester, or tribotester. The prefix "tribo-" indicates a connection between wear, friction, and lubrication. There are probably hundreds of wear testing sets and processes that are well-documented in technical literature and are used regularly in laboratories all over the world. No of the configuration, a wear tester always consists of two pieces that are loaded against one another and move relative to one another. The movement may be generated by a motor or an electromagnetic device. We will use the terms "specimen" and "counter-face" interchangeably to make things easier for readers to understand.[20]

A pin is loaded and forced against a rotating flat disc specimen in a pin-on-disc wear tester to produce a circular wear path. This kind of apparatus is used to evaluate the friction and wear properties of materials in pure sliding circumstances. The counter-face is the other component, whereas the specimen can be either the disc or the pin. The pin's geometry might vary greatly.[21] One useful strategy is to use a ball made of readily available materials as the counter-face, such as bearing steel, tungsten carbide, or alumina (Al_2O_3), giving rise to the term "ball-on-disc." Fig.3.7 shows the set-up used to measure the wear resistance.

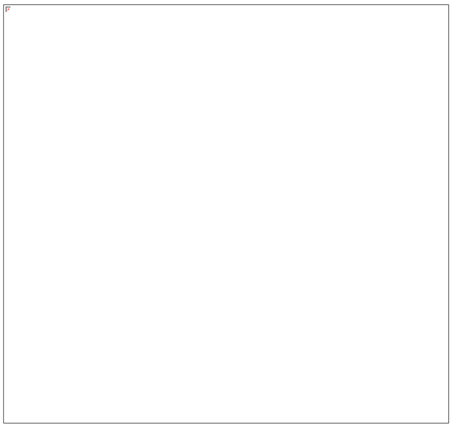

Fig.3.7 Wear Test Set-up

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3.4. Hardness Test

The Vickers hardness test method, also known as the micro hardness test method, is primarily employed for small parts, thin sections, or assessing case depth. This technique relies on an optical measurement system. The micro hardness test procedure, ASTM E-384, specifies a range of light loads, utilizing a diamond indenter to create an indentation that is then measured and converted into a hardness value.[22] This method is highly advantageous for testing a diverse range of materials, but it requires highly polished test samples to accurately measure the size of the impressions. A square base pyramid-shaped diamond is utilized for testing on the Vickers scale. Typically, the loads used are very light, ranging from 10gm to 1kgf. However, "Macro" Vickers loads can exceed 30 kg or even more.

Metals, ceramics, and composite materials can all be tested using micro hardness techniques. The Vickers test is adaptable for a variety of applications thanks to its modest indentation size. It is especially helpful for determining the surface hardness of small components or specialised locations, as well as for testing thin materials like foils. By executing a succession of indentations to produce a hardness profile, the Vickers test is also useful for analysing specific microstructures and evaluating the depth of case hardening. Usually, the part needs to be sectioned in order to suit the tiny specimen size needed for micro hardness testing. Additionally, reliable measurements and a smooth specimen surface can only be obtained with careful sample preparation. Fig.3.8 shows the set-up used to measure the hardness of the prepared samples.

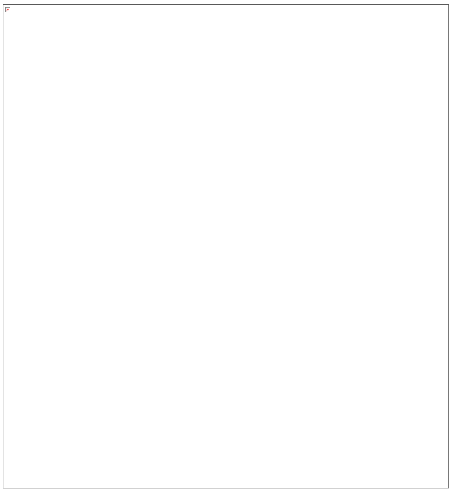

Fig.3.8 Vickers Hardness Test Set-up

3.5. Corrosion Test

Salt spray testing is a corrosion test method designed to expedite the evaluation of coated samples and their suitability as protective finishes. This test exposes the samples to a corrosive environment, simulating the effects of natural corrosion. The main objective is to assess the appearance of corrosion products, such as rust or other oxides, after a specific duration of testing. The test duration varies depending on the corrosion resistance of the coating under evaluation. In general, coatings with higher corrosion resistance require longer testing periods before the onset of visible corrosion. The salt spray test serves as a comparative assessment of different coatings' corrosion performance, Fig. 3.9 shows the set-up used to analyse the corrosion level.

Fig.3.9 Corrosion Test Set-up

4. Results and Discussion

4.1. Wear Analysis

In the case of plain Nickel alloy substrate, it has been observed that the wear loss increases. Specifically, the uncoated material exhibits higher abrasion loss compared to the coated material. This can be attributed to the fact that the wear resistance of the nickel alloy improves when it is coated with Zr. As a result, the wear percentage of the coated material decreases significantly. Table 4.1 provides further details about the significant changes in the wear parameters.

Table 4.1 Wear Analysis for Uncoated and Coated specimens

Material	WearLoss (g)	WearPercentage (%)
UNCOATED	0.032242463	0.17
COATED	0.008031438	0.14

4.2. Hardness Analysis

The hardness of the nickel alloy is being evaluated using a Vickers hardness machine, and the results indicate that the coated nickel alloy is 4 to 5 times harder than the uncoated material. Table 4.2 presents the specific changes in the hardness properties of the material before and after the coating process.

Table 4.2 Hardness Analysis for Uncoated and Coated specimens

Material	HARDNESSINHV@0.5KgfLOAD			
	Hardness1	Hardness2	Hardness3	Average value

UNCOATED	263.5	255.5	249.5	256.2
COATED	1044.2	1018.4	970.6	1011.1

4.3. Corrosion Analysis

The coated Nickel alloy sheet underwent a Salt spray fog test, following the ASTM-B-117-18 standard, which is a corrosion test involving exposure to a salt spray environment. The sample consisted of a nickel-based alloy with a zirconia coating applied on one side. After 24 hours of exposure, the sheet was thoroughly examined on all surfaces, including the flat areas, corners, ribs, and drilled and threaded zones. The coated side of the sheet exhibited no red rust spots and showed no signs of corrosion. However, the uncoated side displayed minimal rusting on the surface. The rust was removed, and the plate was dried and weighed. The weight of the coated nickel-based super alloy was recorded as 30.449g, resulting in a percentage decrease in weight of 0.0197%.

5. Conclusion

The Nickel-based super alloy already possesses excellent wear and corrosion properties, making it well-suited for demanding applications such as engine parts and marine components. Therefore, the nickel-based super alloy serves as an ideal base material. To further enhance its properties, a zirconium coating was applied to the alloy using the Thermal Sprayed Coating process. Several tests were conducted on the coated alloy, including the pin-on-disc test to evaluate wear resistance, the salt spray test to assess corrosion resistance, and the Vickers hardness test to measure hardness values. The following conclusions can be drawn from the results:

- The thermal sprayed coating method successfully applied a zirconium layer to the nickel-based superalloy. The pin-on-disc test revealed that the coated sample exhibited a wear loss of 0.008031438g, while the uncoated sample experienced a wear loss of 0.032242463g. This indicates a significant improvement in wear resistance due to the coating.
- The Vickers hardness test showed that the coated sample had a hardness value of 1011.1HV, whereas the uncoated sample had a hardness value of 256.2HV. This demonstrates that the coated sample has hardness approximately 4-5 times greater than the uncoated sample, making it more suitable for demanding applications.
- The corrosion resistance was evaluated through a salt spray test. After 24 hours of exposure, the coated side of the sheet exhibited no red rust spots on the corners and flat surface, indicating excellent corrosion protection. In contrast, the uncoated side displayed negligible rusting. This confirms that the corrosion characteristics of the coated side are significantly superior to the uncoated side.

In conclusion, the nickel-based superalloy's wear resistance, hardness, and corrosion resistance were all enhanced by the zirconium coating that was applied via thermal spray coating, making it a superb option for demanding applications in the chemical and marine domains.

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