Smart Manufacturing and Material Processing (SMMP)

DOI: http://doi.org/10.26480/smmp.01.2023.48.53

TOPICS IN CHEMICAL & MATERIAL ENGINEERING (TCME)

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RESEARCH ARTICLE

STRUCTURAL EVOLUTION AND PROPERTIES OF CR COATINGS ON RINGS DEPOSITED BY HIGH POWER PULSED MAGNETRON SPUTTERING

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ARTICLE DETAILS

Article History:

Received 20 January 2024
Revised 18 February 2024
Accepted 11 March 2024
Available online 15 March 2024

ABSTRACT

The ring is a key component of the high-speed ring spinning machine, which is in sliding friction environment for a long time. A chromium coating with high hardness is usually prepared on rings surface by electroplating technology to improve the mechanical properties and the working life of rings in the current industrial field. However, the environ-mental pollution associated with electroplating technology has restricted its industrial production, and it is urgent to find a non-polluted coating preparation technology. In this paper, an environmentally friendly high power pulsed magnetron sputtering technology was used to prepare the pure Cr coatings with different thickness on the ring surface. The mi-crostructure, deposition rate, adhesion between film and substrate and tribological property of coatings were character-ized by different means. The results showed that with the prolongation of deposition time, the degree of crystallization of coatings was gradually enhanced, and the surface microstructure was gradually transformed from small and dispersed irregular polygonal structure to circular arc irregular cellular structure. When the deposition time were 120 mins, the film thickness was about 6.4 μm, and the average and instantaneous deposition rates of Cr coating were 53 nm/min and 267 nm/min, respectively The coating had the best film-substrate adhesion, low friction coefficient of 0.41 and the best wear resistance (mass wear rate of 1.8×10-4 g/s).

KEYWORDS

High power pulsed magnetron sputtering, Cr coatings, microstructure, tribology

1. INTRODUCTION

The ring spinning frame is currently the most commonly used equipment in the spinning industry, and the steel ring and steel wire ring are the key components for twisting and winding in the ring spinning frame (as shown in Figure 1). The degree of coordination between the two directly determines the quality of the yarn. The spinning speed directly determines the economic benefits of the enterprise. The spinning speed of the spinning machine is increasing year by year, and the rotation speed of the steel wire ring in the steel ring runway is also increased to 40m/s. However, high-speed inevitably leads to an increase in the wear of the steel ring and a shortened lifespan, ultimately affecting the efficiency and quality of spinning (Lv, 2018; Zu et al., 2010; Guo, 2009). Therefore, reducing wear and prolonging service life of steel rings is the key to the rapid development of the high-speed spinning industry.

In recent years, high-power pulse magnetron sputtering technology has received widespread attention in the field of surface modification due to its advantages of energy conservation, environmental protection, flat surface preparation of coatings, and strong adhesion (Xiong et al., 2020; Kundangar et al., 2022; Zhang et al., 2016; Sameer et al., 2016). However, there are currently few studies on the surface modification of steel ring devices on ring spinning machines using high-power pulse magnetron sputtering technology. Therefore, in this experiment, high-power pulse magnetron sputtering technology was used to prepare pure Cr coatings with different thicknesses on the surface of GCr15 steel sheets used in steel rings. The crystal structure and microstructure of the prepared pure Cr coatings were examined using X-ray diffraction (XRD) and scanning electron microscopy (SEM), and the film based bonding strength of the coatings was measured using a coating adhesion scratch instrument, Characterizing the tribological properties of the coating using a friction and wear testing machine, studying the differences in microstructure, film based bonding strength, and tribological properties of Cr coatings with different thicknesses prepared by high-power pulse magnetron sputtering, and determining the optimal process and thickness for preparing Cr coatings with dense structure, good film based bonding strength, and wear resistance, analyze and discuss the feasibility of using high power pulse magnetron sputtering technology instead of electroplating technology to prepare Cr coatings on the surface of steel rings.

2. EXPERIMENTAL MATERIALS AND METHODS

The experiment used high-power pulse magnetron sputtering technology for Cr coating deposition, with a vacuum chamber size of Φ 450mm × H400mm, the cathode target material is selected as a planar circular pure metal Cr target with a purity of 99.9% and a size of Φ 170mm × H7mm), the working gas is high-purity argon. This experiment uses a high-power pulse power supply to provide a single target with a power of 4kW. The target voltage is 600V, the frequency is 50Hz, the pulse width is 4ms, the substrate bias voltage is -65V, and the bias duty cycle is 80%.Two types of substrate samples, GCr15 polished circular plate and P-type (100) Si plate, were selected for the steel ring. The GCr15 polished circular plate sample was mainly used for testing the single stroke scratch and friction wear performance after the experiment, while the P-type (100) Si plate sample was mainly used for testing the microstructure and thickness of the Cr coating after the experiment. Before the experiment, the substrate was placed in an alcohol solution for ultrasonic cleaning for 20 minutes, then dried with nitrogen gas and installed on the workpiece frame in the

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	Website: Website: www.topicsonchemeng.org.my	**DOI:** 10.26480/smmp.01.2023.48.53

Cite The Article: Chao Yang, Juan Hao, Baichuan Wang, Yuhang Ding, Jing Zhang, Dan Dong, Xiaoyan Wang, BaiLing Jiang, Shaikh Mohammed Ibrahim (2023). Structural Evolution and Properties of Cr Coatings on Rings Deposited by High Power Pulsed Magnetron Sputtering. *Topics In Chemical & Material Engineering, 2(1): 48-53.*

vacuum chamber, with a target base distance of 150mm.During the deposition process, the substrate surface was first cleaned by argon ion bombardment for 20 minutes before the formal deposition of Cr coatings. The Ar flow rate was 60sccm, and the working pressure was maintained at 0.6Pa. Four sets of Cr coatings were prepared under deposition time conditions of 60, 90, 120, and 240 minutes, respectively.

The phase structure of the coating was analyzed using an XRD-7000s X-ray diffractometer (test condition: Cu target K α Line, λ= 0.1542 nm, grazing angle 2°, step size 0.02°, scanning speed 8°/min, scanning range 30°~80°.The surface and cross-sectional morphology of the coating were observed using a JSM-6700F scanning electron microscope, and the coating thickness was characterized by the "average of multiple points on the cross-section" method. The average and instantaneous deposition rates of the coating were calculated based on the thickness, total deposition time, and effective deposition time, respectively. The WS-2005 coating adhesion automatic scratch instrument was used and combined with a metallographic microscope to analyze the single stroke scratch behavior of the coating (the scratch needle is a diamond cone with a cone angle of 120° and a top radius of 0.2mm, with a load of 0-60N).Use a ball disc friction and wear testing machine to test the friction coefficient of the coating and characterize the wear resistance based on the mass wear rate (testing conditions: friction pair Φ6mm GCr15 steel ball, load 2N, test temperature 20±3℃, test time 30 minutes.

Figure 1: The schematic diagram of the ring and traveler

3. EXPERIMENTAL RESULTS AND ANALYSIS

3.1 Phase Structure of Cr Coating

Figure 2 shows the XRD patterns of four sets of Cr coatings. It can be seen from the figures that the Cr coatings on the surface of the steel ring prepared at different deposition times all grow preferentially on (200) crystal planes, belonging to a single-phase Cr structure. As the deposition time increases, the diffraction peak intensity gradually increases, indicating that the degree of crystallization of the coating gradually increases. The average grain size of the coating was calculated using the Scherrer formula, and the results were 23, 27, 35, and 41 nm, respectively, all belonging to the category of nanocrystals (Anders, 2010).

3.2 Microscopic Morphology of Cr Coating

Figure 3 shows the surface SEM morphology of the Cr coating. It can be seen that when the deposition time is 60 minutes, the surface of the coating exhibits small and scattered irregular multi angular particle morphology. With the continuous increase of deposition time, the surface particle size of the coating increases and presents a triangular pyramid shape. As the deposition time increases to 240 minutes, the surface particles of the coating exhibit an irregular cellular structure with arc-shaped characteristics. Figure 4 shows the cross-sectional morphology of the Cr coating, which shows that the Cr coating grows in a columnar structure, but the tendency towards columnar growth gradually weakens (Bonzin et al., 2008). As the deposition time continues to increase, the size of the columnar structure of the coating significantly increases, and the voids and gaps between the columnar structures gradually decrease, indicating an improvement in the density of the coating.

Figure 5 shows the corresponding relationship between the thickness of Cr coating and deposition rate. Combined with Figure 4, the thickness of the coating can be measured as 4.02, 5.20, 6.40, and 11.68μm respectively. The average deposition rates of the corresponding coatings are 67, 58, 53, and 49 nm/min. Due to the small duty cycle of high power pulsed electric fields, the actual deposition time of the coating is much shorter than the total process time. The instantaneous deposition rates of the coating were calculated as 335, 288, 267, and 243nm/min, respectively. The coating has a high instantaneous deposition rate, mainly due to the high power pulse electric field being able to obtain extremely high instantaneous target power, greatly improving the deposition rate of the coating. As the deposition time increases, the deposition rate of the coating decreases, indicating a decrease in the increment of coating thickness. This is mainly because as the thickness of the coating increases, the internal stress inside the coating gradually increases, and during the coating deposition process, the phenomenon of coating stress peeling occurs, leading to a decrease in the coating deposition rate.

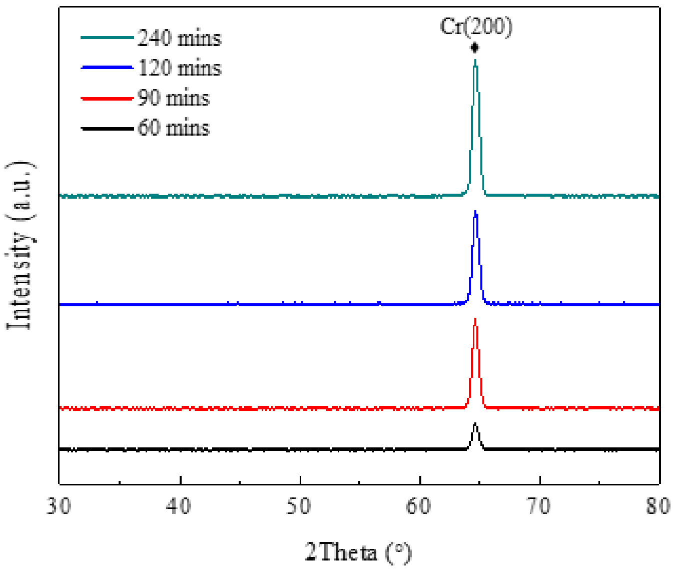

Figure 2: XRD patterns of Cr coatings

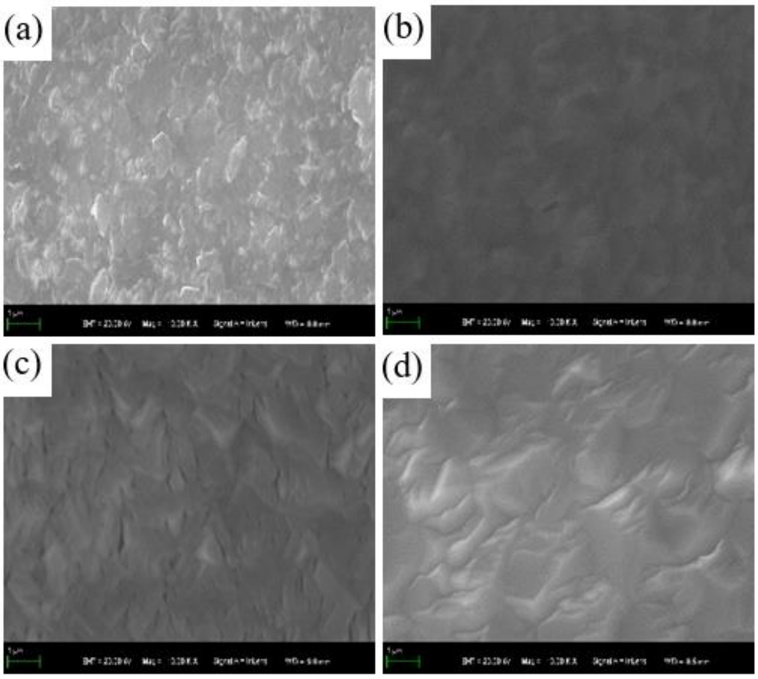

Figure 3: Surface morphologies of Cr coatings deposited under different deposition time (a)60 mins; (b)90 mins; (c)120 mins; (d)240 mins

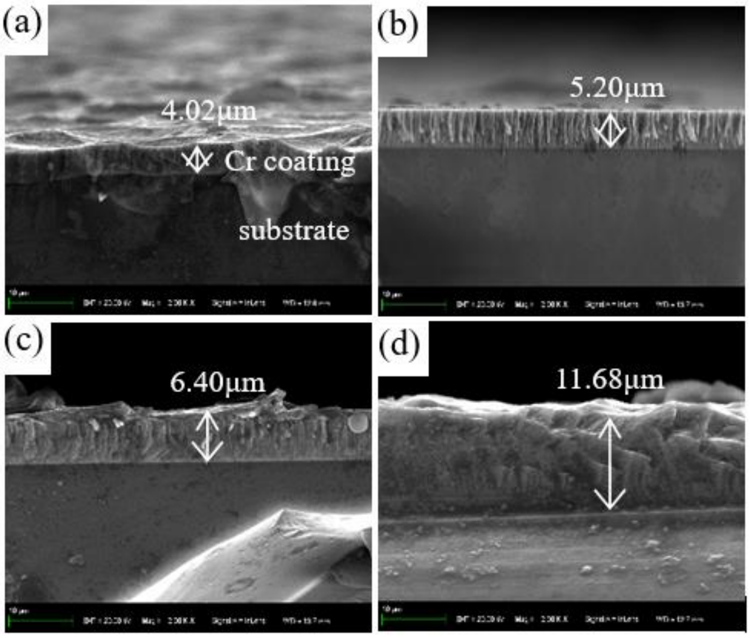

Figure 4: Cross-section morphologies of Cr coatings deposited under different deposition time (a)60 mins; (b)90 mins; (c)120 mins; (d)240 mins

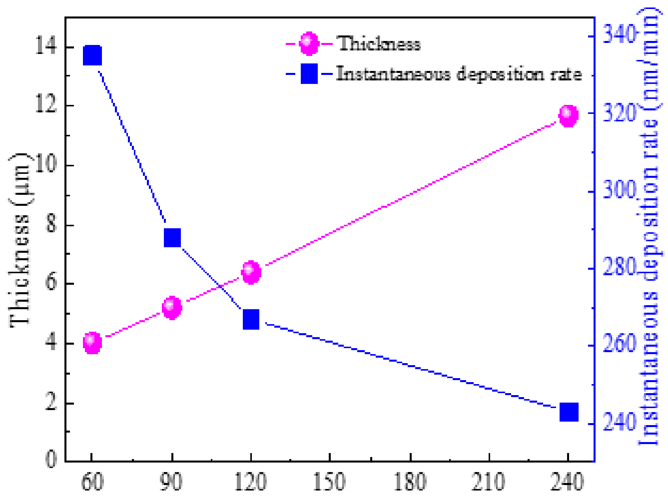

Figure 5: The thickness and instantaneous deposition rate of Cr coatings deposited under different deposition time

3.3 Single Row Scratch Behavior of Cr Coating

Figure 6 shows the single-row scratch morphology photos of Cr coatings prepared at different deposition times. When the deposition time is 60 minutes, the coating begins to peel and detach in a large area under a small load (about 5.1 N).The scratch failure mechanism is mainly manifested as peripheral peeling (Hovsepian et al., 2006). When the deposition time is 90 minutes, the indenter has a squeezing effect on the coating on both sides of the scratch groove, and continuous microcracks appear inside the scratch groove. With the increase of loading load (about 22.5N), small areas of peeling appear at the tail of the scratch, and the scratch failure mechanism is mainly manifested as peripheral peeling. When the deposition time is 120 minutes, the interior and edges of the scratch groove are relatively smooth, and there is no obvious peeling or detachment of the coating. When the deposition time increases to 240 minutes, under the compression of the indenter, the coating undergoes severe uplift and detachment under relatively small loads (about 11.8N), and the detachment area is approximately circular in shape. The scratch failure mechanism is mainly manifested as compression peeling (Purandare et al., 2010).

3.4 Friction and Wear Properties of Cr Plating

Figure 7 shows the friction coefficient curves of Cr coatings prepared at different deposition times. The Cr coating deposited at different deposition times undergoes a brief running-in period during the friction and wear process, gradually entering a gentle stage and eventually reaching a stable stage. The average friction coefficient during the stable stage is used to characterize the friction coefficient (Yang et al., 2015). As shown in the figure, with the continuous increase of deposition time, the average friction coefficient of the coating shows a trend of first slightly decreasing and then significantly increasing. When the sedimentation time is 90 minutes, it reaches its lowest point, approximately 0.38. Figure 8 shows the wear rate of Cr coatings prepared at different deposition times. With the continuous increase of deposition time, the mass wear rate of the coating shows a trend of first decreasing and then increasing. When the deposition time is 120 minutes, the wear rate is the smallest (1.8×10-4g/s), indicating that this coating has the best wear resistance

Wear is an inevitable result of friction, which is the process of continuous loss of the contact surface between two relatively moving objects when they come into contact. The wear resistance of the coating is closely related to its structural composition, density, hardness, and film based bonding strength (Wen et al., 1993). For the same material, the denser the structure, the higher the hardness, and the stronger the adhesion between the film and substrate, the stronger the ability of the coating to resist wear, that is, the better the wear resistance. From the experimental results, it can be seen that the microstructure of the coating becomes denser with the prolongation of deposition time. However, due to the poor film-based bonding strength of the coating prepared at 240 minutes, the Cr coating prepared at 120 minutes has high wear resistance.

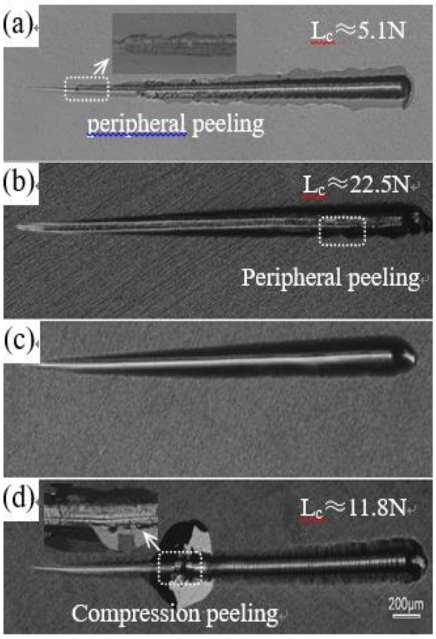

Figure 6: The scratch morphology of Cr coatings (a)60 mins; (b)90 mins; (c)120 mins; (d)240 mins

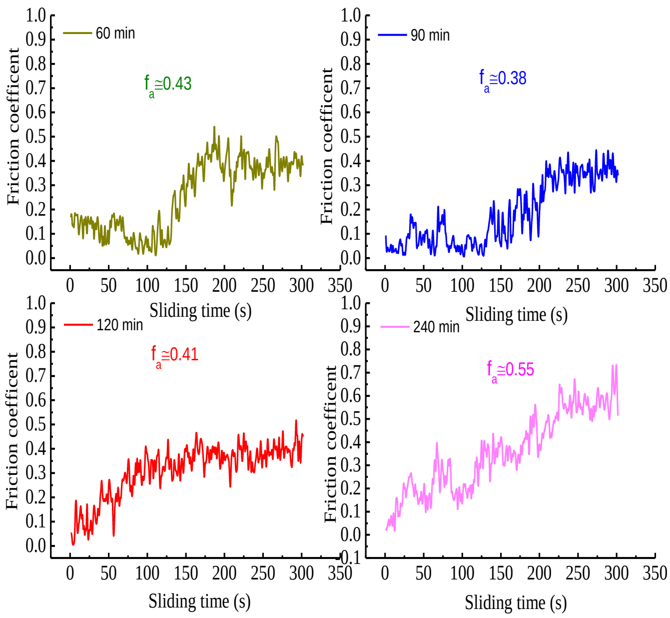

Figure 7: The friction coefficient of Cr coatings deposited under different deposition time

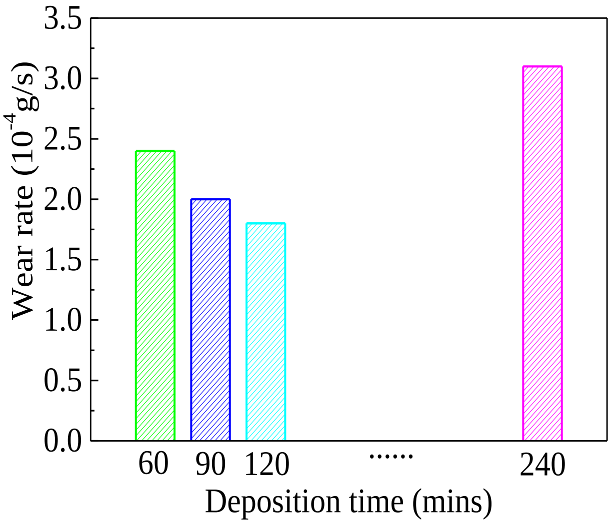

Figure 8: The mass wear ratio of Cr coatings deposited under different deposition time

3.5 Analysis and Discussion

Both electroplating and high-power pulse magnetron sputtering techniques can prepare Cr coatings on the surface of the substrate, but the differences in their preparation principles result in different microstructures and properties of the coatings. The preparation of Cr coating by electroplating technology involves the process of direct current being applied to an electrolyte solution rich in Cr ions, and an electrochemical reaction (redox reaction) occurs on the surface of the cathode substrate to prepare a Cr coating. The growth of electroplated Cr coating meets the classical theory of adsorption diffusion nucleation growth, and grows layer by layer in a layered manner. If the current density, plating solution concentration and other parameters of the electroplating process are reasonably adjusted, the nucleation driving force can be increased and the growth driving force can be reduced, achieving the nanoscale coating and improving the mechanical properties of the coating (Fu et al., 2020). However, during the growth process of the coating, there will be a small amount of pores and a large amount of stress generation, resulting in the appearance of internal voids and surface grid cracks in the coating, which affects the wear resistance of the coating.

The preparation of Cr coating by high-power pulsed magnetron sputtering technology is a process of using high-power pulsed electric fields to eject surface atoms of Cr metal targets and deposit them onto the substrate. The deposition process of the coating is a low-temperature deposition process, where the surface diffusion energy of adsorbed atoms on the substrate surface is higher than the bulk diffusion energy, resulting in the coating growing in a fibrous or columnar structure (Liu et al., 2004). High power pulsed electric fields can increase the number and activity of deposited particles, increase the critical nucleation number of the coating, facilitate the obtaining of nanoscale grains in the coating, and also densify the coating structure. Nanoscale grains and dense columnar structures can improve the mechanical properties and wear resistance of the coating. Based on the above experimental results and analysis, it is shown that high-power pulse magnetron sputtering technology can prepare Cr coatings with dense microstructure, good adhesion and wear resistance on the surface of steel rings.

4. Conclusion

1) The surface structure of the Cr coating prepared by high-power pulse magnetron sputtering gradually transforms from a fine and dispersed multi angular granular morphology to an irregular cellular morphology with arc-shaped characteristics, and grows preferentially along the (200) crystal plane. With the continuous increase of deposition time, the crystallization degree of the coating increases, all of which belong to nanocrystals.

2) With the continuous increase of deposition time, the instantaneous deposition rate of the coating shows a slow downward trend, with the highest average deposition rate and instantaneous deposition rate reaching 67 and 335nm/min, respectively, which can achieve rapid preparation of the coating.

3) When the deposition time is small, the failure mechanism of the coating scratch is manifested as peripheral peeling; When the deposition time is large (240 minutes), the scratch failure mechanism of the coating is manifested as compression peeling. When the deposition time is 120 minutes, there is no significant peeling or detachment of the coating; Meanwhile, its average friction coefficient is only 0.41, and its mass wear rate is the smallest (1.8 × 10-4g/s), the adhesion strength and wear resistance of the membrane substrate are both good.

Acknowledgements

This work is financially supported by National Natural Science Foundation of China (No. 52001251), Xi'an Science and Technology Plan Project (23GXFW0055), China Post-doctoral Science Foundation (No.2021M692597), General project of Education Department of Shaanxi Provincial (21JK0799) and Natural Science Basic Research Project in Shaanxi Province (2022JQ-556), Shaanxi Provincial Natural Science Basic Research Program Project (No. 2024JC-YBQN-0525). We thank the reviewers for their informative and insightful suggestions regarding our work.

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