Research on Engineering Structures & Materials
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Research Group

Research Article

Enhancing the corrosion resistance and potential biomedical applications of titanium dental implants coated with niobium pentoxide

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Article Info

Article History:

Received 11 Oct 2025

Accepted 27 Dec 2025

Keywords:

Biocompatibility;
Corrosion resistance;
Titanium;
Dental implants;
Spin coating,
Niobium pentoxide

Abstract

The excellent mechanical strength and clinical reliability of titanium-based implants are key advantages of their applications in biomedical fields. However, their susceptibility to bacterial colonization and corrosion remains a major limitation, affecting long-term performance and compromising patient safety. The present study described a niobium pentoxide coating, applied using the spin coating technique followed by thermal annealing, as a viable surface modification approach that can be used to enhance the corrosion resistance and antibacterial performance of titanium dental implants. The Nb_2O_5 suspension was prepared under controlled conditions and subsequently deposited, in multiple layers, onto the substrate to form a uniform protective film. Electrochemical characterization by potentiodynamic polarization (Tafel analysis) demonstrated a significant decrease in the corrosion rate, from 0.0077 mm/year for uncoated to 0.0021 mm/year for coated titanium, representing a ~73% decrease in corrosion current density. These results proved that a stable passive layer had formed, enhancing surface stability in physiological media. Successful crystallization of Nb_2O_5 phases upon thermal treatment, as confirmed by XRD analysis, accounted for the enhanced electrochemical performance. Antibacterial properties against Staphylococcus aureus ATCC 25923 were evaluated by the agar diffusion method. Results obtained showed that the diameter of the inhibition zone had increased from 12.9 ± 0.4 mm (uncoated) to 15.1 ± 0.5 mm (coated), corresponding to a statistically significant improvement of 16.7% ($p < 0.05$). Enhanced antibacterial properties were related to changes in surface properties owing to the deposition of Nb_2O_5 films, leading to reduced bacterial adhesion and biofilm growth. Conclusion The Nb_2O_5 coating contributed significantly to the improvement of corrosion resistance and the antibacterial properties of the titanium implants, hence demonstrating biomedical relevance for future applications in dentistry.

1. Introduction

When it comes to metals, titanium and its alloys are considered the standard due to their mechanical properties, resistance to corrosion, and biocompatibility [1]. Their resistance to oxidation due to the formation of stable oxide, i.e., TiO_2 layer at the surface make them endure the stress of oral environment with wide ranging pH, presence of microbial biofilms, and cyclic mechanical loads. Even

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DOI: http://dx.doi.org/10.17515/resm2026-1238ic1011rs

with these benefits, titanium implants have their limitations. However, complications such as peri-implantitis, ion release, and insufficient osseointegration are still the main problems, which may cause peri-implantitis and conclusion of implant failure. The novelty in this work was the rational use of niobium pentoxide (Nb_2O_5) for modifying, in a single action, the surface of titanium implants to improve their corrosion resistance and to make them antibacterial. Although titanium is commonly used in medical implants, its natural ability for the adhesion of bacteria and its susceptibility to degradation upon exposure to physiological environment requires the development of surface modification methods. The choice of Nb_2O_5 was prescribed by its inherently known chemical stability, as well as its known osteointegrative and antimicrobial properties. In contrast to traditional coatings targeting only a single limitation, this dual-functional strategy is expected to combat two important limitations (biofouling and electrochemical degradation) simultaneously and thus provide a new solution to improvement of the longevity and safety of implants. The decision to pursue this direction was further supported by the growing demand for multifunctional implant surfaces capable of addressing postoperative complications without compromising mechanical performance.

A significant challenge is the potential for bacterial biofilm development on the surface of dental implants, leading to chronic infections (Peri-implantitis) and implant failure. Peri-implantitis, an inflammatory condition induced by bacterial biofilms, contributes to the risk of implant failure and represents a major risk to long-term implant success. Moreover, titanium ions release in a corrosive oral environment raises concerns about possible produced cytotoxic and inflammatory responses, compromising both patients' safety and implants service life. However, these problems require novel approaches to improve the surface properties of titanium implants [2]. Silver nanoparticle coatings on titanium to reduce adhesion of bacteria These coatings not only killed bacteria but were also cytotoxic to osteoblasts, and therefore were not suitable for clinical application [3].

Olivares-Navarrete al. [4] tested the biocompatibility of niobium and its oxides using in vitro cell culture testing. Nb_2O_5 showed good chemical stability and biocompatibility, and was identified as a potential promising material for biomedical anode [5]. Nascimento et al. [6] sprayed Nb_2O_5 coatings on titanium to study their antibacterial activity. They also demonstrated that Nb_2O_5 strongly suppressed bacterial growth by indirect rather than direct manners without toxicity to human cells, suggesting its application as safe and efficient implant coatings [7].

Bioactive coatings have attracted attention in recent years to enhance the functional performance of titanium implants, facilitated by the development of surface modification technologies. Notably, metallic oxides have been well explored to overcome this challenge, focusing on the twofold property of enhanced corrosion resistance and increased biocompatibility. Niobium pentoxide (Nb_2O_5) has been investigated as a promising material due to its high chemical stability, its non-toxicity and the formation of a dense protective oxide sulhouette. Furthermore, Nb_2O_5 coatings have been reported to display antimicrobial properties and to enhance osseointegration by promoting cell adhesion and proliferation. In the field of biomedical applications, niobium-based coatings have been researched in several studies. For instance, Pakshir et al. investigated the tribo-corrosion characteristics of Nb_2O_5-coated titanium and found a remarkable improvement in mans of corrosion resistance and wear in simulated body fluids. In another study conducted by Gutwein and Webster (2004), they investigated the impact of nanostructured niobium oxide on cell attachment by demonstrating that the nanosized characteristics of the coating enhance osseointegration through osteoblast adhesion and proliferation. Sathish Kumar et al. also attempted. confirmed that the Nb_2O_5 coating had antibacterial activity against Staphylococcus aureus and Escherichia coli, indicating the potential ability of Nb_2O_5 to reduce the likelihood of implant-associated infections [8].

Although niobium pentoxide shows promising results in the previously performed studies, there are only few studies related to niobium pentoxide in the dental field and still fewer with titanium implants. Recognizing this fact, the aim of this research study was to present a pure Ti alloy and to describe the advantages that this alloy could have in bio applications, including improved corrosion resistance, reduced ion release, and improved enhancements in biocompatibility. The objective of this work is to assess the potential of niobium pentoxide coatings to enhance the biocompatibility and corrosion resistance of titanium dental implants. This study aims to bridge this gap and develop more stable and clinically relevant dental implants systems incorporating the inherent drawbacks of traditional titanium implants.

2. Materials and Methods

2.1. Materials

Titanium samples were used. Pure titanium rod grade ASTM F67 GR2 was purchased from Shaanxi Yunzhong Industry Development CO., LTD. It is a 1000 mm length and 10 mm diameter rod; the rod was cut into 25 samples using a wire cutting machine, each with dimensions of 10 mm in diameter and 10 mm in length, as shown in table 1.

Table 1. Chemical composition of titanium

#	Material	Fe	C	N	O	H	Ti
1	Ti	0.3	0.08	0.03	0.25	0.015	remainder

2.2. Surface Treatments

All samples were ground using silicon carbide (SiC) papers in the following series (400, 600, 800, 1000, 1200, and 2000 grain sizes) to achieve a clean and flat surface, after which the samples were polished to prepare a clean, uniform, smooth surface for the coating processes. to verify that the surface property and the remainders of it are the same in every example. Given before, offer better adhesion and performance of the coatings [9].

2.3. Coating Process

Such a coating procedure was done in three steps: preparation of an Nb_2O_5 suspension, spin coating, and thermal annealing. To prepare the suspension, 0.5 g of Nb_2O_5 powder was dispersed in a chemically resistant glass vessel. Then, 10 mL of diluted sulfuric acid (H_2SO_4, 1:1 water-to-acid) was slowly added under stirring to prevent clustering, followed by mild heating in a water bath (10–15 min) to promote dispersion. Subsequently, 10 mL of absolute ethanol was added, and the mixture was stirred until homogeneous. The final suspension had a concentration of 25 mg/mL, a measured pH of 2.0, a density of 0.98 g/mL, and a dynamic viscosity of 3.2 mPa·s at 25 °C. The suspension was aged for 24 h at room temperature prior to coating. For spin coating, 2–3 drops (~60–90 μL) of suspension were dispensed onto the pure titanium substrate; the sample was first spun at 800 rpm for 10 s to ensure uniform spreading and then at 4000 rpm for 45 s to obtain a thin and smooth layer. Three layers were deposited, with intermediate drying at 90 °C for 10 min between layers. Thermal annealing was performed in an argon atmosphere at 600 °C for 1 h, with a heating rate of 5 °C/min. This temperature was chosen to ensure Nb_2O_5 crystallization and coat adhesion. The inert atmosphere prevented oxygen diffusion into the titanium substrate and thereby avoided the formation of alpha-case. The post-annealing SEM and electrochemical analysis confirmed that the integrity of the titanium substrate was preserved without its embrittlement or degradation. [10-12]

2.4. Characterization Methods

X-ray diffraction (XRD) (Shimadzu, 6000) using room temperature with a scanning rate of 5/min Cu Ka radiation (λ=1.5405 A°) and applied energy (40 kV and 30 mA) and consequently scanning electron microscope (SEM) (FEIQUANTA 250, Czech Republic) to prove the particles form and to estimate its thickness.

2.5. Corrosion Test

Corrosion tests were performed on the samples before and after the coatings were applied. performed tests like open circuit potential (OCP), Tafel analysis, cyclic polarization, and electrochemical impedance (EIS). The Corrtest Instruments Potentiostat/Galvanostat BS-21 device, in a 37°C at pH 7 system. The corrosion media used is Ringer's Solution [13]. The formula for the Experiments Corrosion Rate (CR) is as follows (CR = (K × Icorr) / (ρ × A), where K is a constant (0.00327), Icorr is the corrosion current density (A/cm^2), ρ is the density of the material (g/cm^3), and A is the area of the sample (cm^2).

3. Results and Discussion

3.1 XRD Analysis of Titanium Before and After Niobium Pentoxide (Nb_2O_5) Coating

X-ray diffraction analysis was made to determine the phase composition and structural parameters of the uncoated titanium and Nb_2O_5-coated titanium. Figure 1 shows the diffraction patterns of both the samples, in which the black spectrum is for pure titanium and the red spectrum is for the coated sample.

The diffraction peaks for uncoated titanium, at 2θ values of 35.32°, 38.56°, 40.36°, 53.12°, 63.12°, and 70.72°, matched the hexagonal close-packed (hcp) structure of α-Titanium, according to JCPDS card no. 44-1294. These sharp and well-defined peaks confirm the crystalline nature of the titanium substrate. After Nb_2O_5 coating and thermal annealing, the additional peaks at 22.96°, 28.68°, 35.44°, 38.72°, 40.52°, 53.32°, 63.24°, and 70.92° correspond to the orthorhombic phase of Nb_2O_5, identified by using JCPDS card no. 30-0873. This indicates that the Nb_2O_5 coating was successfully deposited and crystallized on the titanium surface.[14]

Some degree of peak broadening was perceptible in the coated sample, especially for Nb_2O_5-related reflections. The peak broadening can be ascribed to lattice strain, reduced crystallite size, or partial amorphization within the coating layer. From the FWHM of the most intense Nb_2O_5 peak and assuming that peak broadening is dominated by particle-size effects, the average size of the crystallites was estimated using the Scherrer relationship:

$$D = \frac{K\lambda}{\beta\cos\ \theta} \tag{1}$$

where D is the crystallite size, K=0.9 is the shape factor, λ=1.5406Å is the wavelength of Cu Kα radiation, β is the FWHM in radians, and θ is the Bragg angle. The calculated crystallite size for the Nb_2O_5 coating was approximately 32 ± 3 nm, indicating nanoscale crystallinity.

This resulted in a crystallite size of ~32 ± 3 nm for Nb_2O_5 coating, which showed nanoscale crystallinity. In conclusion, the XRD data confirm the crystalline formation of a Nb_2O_5 phase on the titanium surface, which can be related to the enhancement of corrosion resistance and surface stability of the coated samples. These structural improvements support the coating's biomedical relevance and also its potential for dental implant applications, as depicted in Figure 1.

3.2 Surface Morphology Analysis of Nb_2O_5 Coating Using SEM

Nb_2O_5-coated titanium specimen (the Nb_2O_5 layer is rather uniform, indicating that the spin coating technique is very efficient for producing a thin and adherent homogenous coat). The surface morphology is of nano-structured or micro-structured scale, which is important for better cell adhesion and bio-compatibility. In addition, the nano roughness would improve the osteointegration so that the coating is also useful for dental implantations.

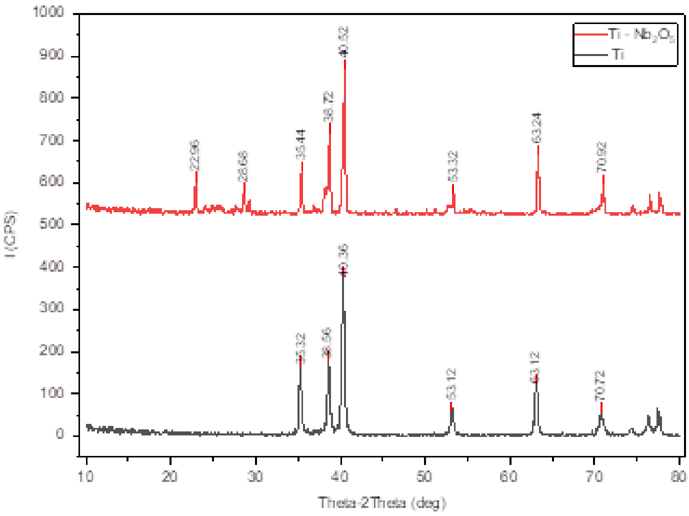

Fig. 1. XRD of Ti and Ti – Coated

The SEM images further visualize the general homogeneity and continuity of the coating, which are important for providing consistent anti-corrosion and bacteria-resistant protection. Any surface cracks or defects are probably caused by the thermal expansion–contraction that occurs during the annealing and likely can affect the long-term durability of a coating. Bacterial can also be prevented from adhesion by any smooth surface, which help to increase the antimicrobial efficiency by controlling the bio film formation [15]. Moreover, the nanopores within the coating surface may facilitate its interaction with biofluids resulting in an enhanced biological response and integration. This can lead to non-uniformities or agglomeration of the coating and local weaknesses may arise that might be addressed by adjusting the deposition parameters (spin, precursor concentration, annealing conditions) so as to give uniform coverage over the entire surface of the substrate [16,17], as depicted in figure 2

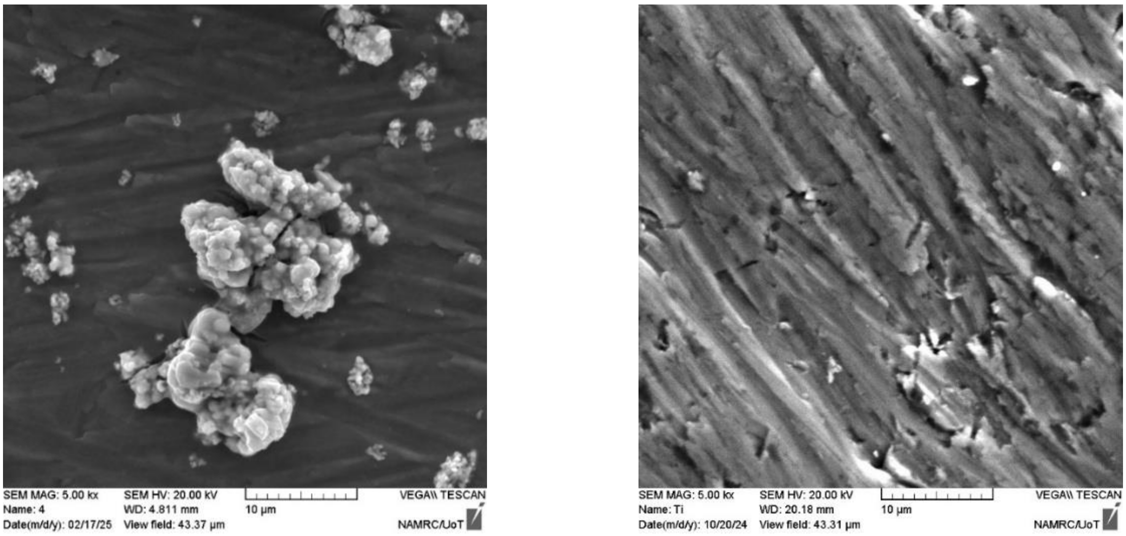

Fig. 2. SEM of Ti – Coated and Pure Ti

3.3 Corrosion Behavior Analysis of Titanium Before and After Coating

The potentiodynamic polarization curves of bare titanium and Nb_2O_5-coated titanium for the electrochemical responses are carried out as shown in Table 2, Figure 3. The test data provided key values that describe the corrosion resistance of the material such as corrosion potential (Ecorr), which is lower than for BA in each but also its icorr, Tafel slopes (bc and ba), CR, Rp on material, and IE (%). [18]. The untreated Ti had Ecorr (−0.054 V vs SCE) that was more noble than the coated sample (Ecorr: −0.108 V vs SCE). The positive shift in E_corr indicates an enhanced thermodynamic stability and a decreased likelihood of spontaneous corrosion. Therefore, it is proposed that Nb-oxide coated titanium has long term stability of the surface. [19]

The icorr, which reflects the rate of metal dissolution, decreased significantly from 6.58×10^{-7} A/cm^2 for uncoated to 1.78×10^{-7} A/cm^2 for coated sample. This ~73% decrease in icorr guarantees the barrier role of Nb_2O_5 layer due to retarding the charge transfer and partially blocking corrosive substance from contacting. [20] Similarly, the Tafel slope values also varied significantly between coated and uncoated samples. The cathodic slope, on the other hand, was enhanced from 29.846 mV/dec to 40.385 mV/dec and the anodic slope was increased from 34.983 mV/dec to 43.348 mV/dec. These changes are also evidence for a shift in electrochemical kinetics likely associated with enhanced passivation and reduced active surface area in contact with the electrolyte.

The CR value that was converted from icorreng into mm/year, decreased with coating (0.00209 mm/year) compared to uncoated titanium (0.00772 mm/year), this great decrease supports the good protection that the coating gives to the substrate under simulated physiological conditions. The polarization resistance Rp increased from 1.04×10^5 Ω·cm^2 to 4.38×10^5 Ω·cm^2, proving the high resistance for electrochemical reaction. In addition, an inhibition efficiency of 73% was calculated for the Nb_2O_5 coating, and this further validated its protection.

Figure 3 shows the Tafel plots of both samples, indicating an obvious shift of corrosion potential to the right and a decrease in current density for the coated titanium. The plot of the coated sample is flatter, with slower kinetics of corrosion and improved stability on the surface. In short, the Nb_2O_5 coating decisively improves the corrosion resistance of pure titanium by decreasing icorr and CR, increasing Rp, and enhancing %IE. The substantial improvements are related to the growth of a stable, adherent, and passivating Nb_2O_5 layer acting as a physical and chemical barrier against aggressive ions. Such enhancements are very important for extending the lifespan and improving biocompatibility of titanium dental implants in the corrosive environment of the oral cavity. [21-23]

Table 2. Corrosion parameters of Titanium

	-Ecorr	icorr	-bc	+ba	CR	R_p	%IE
	(mV)	($A.cm^{-2}$)	$mV.dec^{-1}$		(mm/a)	Ω·cm2	%
Ti	0.054154	0.000000658070	29.846	34.983	0.00772472938461539	104,000	
Ti- Nb_2O_5	0.10797	0.000000177670	40.385	43.348	0.00208557246153846	438,000	73%

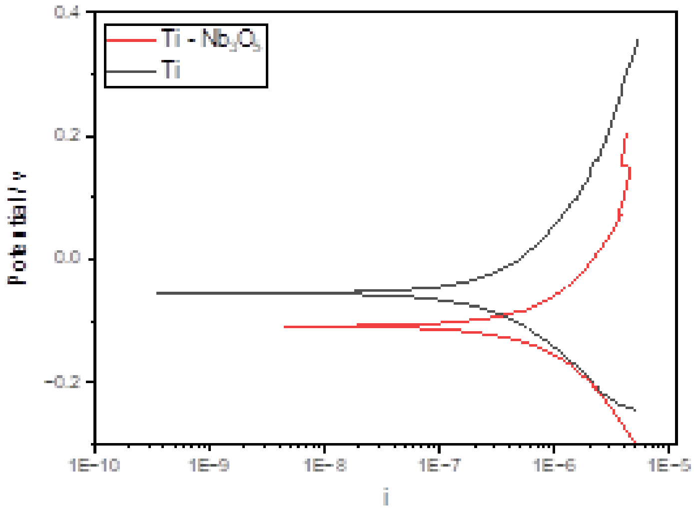

Fig. 3. Electrochemical behavior of Ti and Ti - coated

3.4 Inhibition Zone Diameter Results

The antibacterial activity of pure titanium and Nb_2O_5-coated titanium was measured by the agar diffusion method against Staphylococcus aureus ATCC 25923 using Mueller–Hinton agar plates that were inoculated with bacterial suspensions at 1×10^6 CFU/mL (0.5 McFarland standard) under an aerobic atmosphere at 37 °C for 24 h. The inhibition zones were measured in triplicate and expressed as mean ± SD. The uncoated titanium showed an inhibition zone diameter of 12.9 ± 0.4 mm, while Nb_2O_5-coated titanium exhibited a higher diameter of inhibition zone to 15.1 ± 0.5 mm (n = 3).

3.5 Rise in Antibacterial Activity

Compared to uncoated titanium, the diameter of the inhibition zone was higher by 16.7% for the coated sample, and the increase was statistically significant ($p < 0.05$). From this result, it may be assumed that Nb_2O_5 coating increases the antibacterial properties of titanium. Presumably, a stable oxide layer is formed, which reduces bacterial adhesion and biofilm formation. Niobium pentoxide is reported to be a biocompatible material with the ability to alter surface properties in a way that is not favorable for microbial colonization. Thus, the increased diameter of the inhibition zone was attributed to surface modifications induced by the coating, which alter the kinetics of bacterial adhesion and create a less favorable environment for bacterial growth. [24,25]

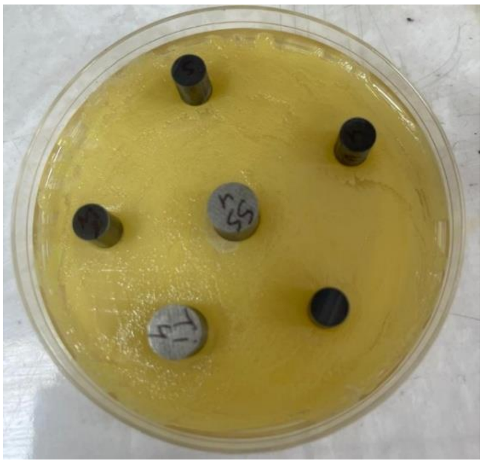

Fig. 4. Inhibition Zone Test of Ti and Ti - coated

4. Conclusions

The corrosion and antibacterial properties of pure titanium substrates coated by Nb_2O_5 using spin coating followed by thermal annealing have been considerably improved. Through electrochemical characterization, a great decrease in corrosion current density and corrosion rate was proved, while a great increase in polarization resistance was obtained, suggesting the development of a steady, protective passive layer. By XRD analysis, the successful crystallization of Nb_2O_5 phases contributing to the enhanced electrochemical stability of the coated samples was verified. Antibacterial testing against Staphylococcus aureus resulted in a statistically significant widening of the diameter of the inhibition zone, which verifies the ability of this coating to diminish bacterial adhesion and biofilm growth.

These findings emphasize the biomedical relevance and possible clinical applications of Nb_2O_5-coated implants made of titanium, especially in dentistry, where long-term durability and resistance to microbial colonization are important. Although direct cytotoxicity or biological compatibility tests were not shown in this work, these observations on the enhancement of corrosion resistance and antibacterial activity clearly suggest that Nb_2O_5 coatings have excellent potential for prolonging the service life and reliability of titanium-based implants. Further work is recommended, including biological and cytotoxicity evaluations to establish coating performance in vivo.

Acknowledgement

The authors extend their heartfelt appreciation to the Nanotechnology and advance materials Research Center/University of Technology, for their support and the facilities provided during the course of this research. Thanks, and appreciation goes to the faculty members and technical staff for their invaluable assistance with the corrosion, XRD, and antibacterial tests. They have been helpful during this research as in the long run this research would not have reached completion without their guidance and cooperation.

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