# INFLUENCE OF SUBSTRATE SURFACE ROUGHNESS ON THE THERMAL EMISSIVITY OF TITANIUM CARBIDE COATINGS ON GRAPHITE

#### by

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This study focused on the impact of substrates shape on the heat radiationcharacteristics of a coating made of titanium carbide, TiC, deposited over a graphite basis. The TiC coating emissivity increase by 29.65% at 1050 °C and by 37.45% at 1650 °C when graphite, substrate surface roughness, was decreased from 3.01  $\mu$ m to 0.73  $\mu$ m. Simultaneously, the TiC coating's spectrum emissivity on the graphite substrate indicated the material's clear characteristic heat radiation. These findings demonstrated that the coating and substrate interacted to determine the coating's heat radiation properties. A simplified coating model created to consider how the shape of the substrate affects the coating's ability to conduct heat. Ultimately, the rough form of the substrate led to a decrease in the coating's heat radiation characteristics and an enhancement in energy loss at the interface.

Key words: heat radiation, emmissivity, graphite, substrate, temperature, TiC

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## Introduction

The TiC, is a vital ceramic substance used in many different military and civilian applications [1]. It is a highly desirable material due to its high specific strength and modulus as well as its low thermal expansion and strong thermal conductivity. In applications like solar cell film, wear-resistant coating, anti-oxidation coating for preservatives, and so forth, coatings or films are crucial forms of TiC [2]. The TiC coatings are frequently used in high temperature environments when in use, for as in industrial heat exchangers or thermal protection systems used in aircraft and spacecraft [3]. To prevent oxide etch in passive heating systems like thermal protection systems, TiC coating is frequently used as the layer of barrier. In this situation, heat is transferred through the coating to the underlying material [4].

Heat is transferred from the graphite components to the TiC coating, with the flow direction being from the substrate to the coating. The heat transmission properties of a coating are influenced by a number of factors, including the coating itself, the substrate, the interface's thermal resistance, and the coating's proximity to other heat sources [5]. Notably, at elevated temperatures, heat radiation becomes the main mechanism governing the exchange of heat between materials and their environment.

The TiC emits an electromagnetic field due to the random thermal mobility of its electrons, which is known as heat radiation. A material's infrared emissivity is a key property for defining its heat radiation properties [6]. A substance's emissivity could be calculated by contrasting its own emission intensity with that of a black body. The Stefan-Boltzmann equation states that heat radiation rises rapidly with temperature, particularly at maximum temperatures [4, 7].

So, the heat radiation attributes of a TiC coating substantially affect heat transfer and the temperature distribution across components. When the substrate and coating share identical chemical compositions and thicknesses, it is their respective morphologies that most significantly influence the coating's heat radiation efficiency [8]. Heat radiation correlations with coating surface features such roughness, existence of an oxide layer, and contaminants have been the subject of a large body of theoretical and empirical research. The influence of surface shape on emissivity has been demonstrated using a number of models. To illustrate the connection between surface roughness and emissivity, for instance, the researchers presented an equation and a model for approximation [9].

The analysis of absorptivity and emissivity in the context of surface roughness, with feature sizes comparable to the wavelength of interest, has been categorized into three distinct regimes. For ceramics composed of  $ZrB_2$  with 15% TiC, the machined surface pattern has been shown to considerably influence heat radiation at temperatures below 1550 K. A number of mathematical models have also been developed to articulate the correlation between the surface morphology and its heat radiation properties [10]. Non-etheless, a dearth of research has examined how the substrate affects emissivity when examining the coating's heat radiation characteristics. There was a significant degree of interference at the substrate-coating interface when heat flowed from the substrate to the coating. Related studies have demonstrated that substrate morphologies and composition significantly influence the coating's heat radiation when coating thicknesses range from a few microns to tens of microns [11].

Using a Ni alloy substrate, researchers [12] studied how changing the substrate shape affected the heat radiation parameters of an Au film. The findings demonstrated that raising the substrate roughness led to the destruction of Au film integrity in addition an increase in effective surface emitting area [13]. The majority of studies disregarded the substrate's impact on the coating's characteristics related to heat radiation. In the current work, chemical vapour

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deposition (CVD) was used to create a TiC coating on three distinct morphologies of graphite substrates. Researchers looked into how substrate morphology affected heat radiation characteristics measured from the TiC coating. There was also discussion of the substrate-coating heat transmission methods. To attempt a detailed analysis of the physical phenomena involved, a basic model had been developed.

# Materials and methods

### Preparation of material

The high purity graphite was sliced on a substrate measuring  $10 \text{ mm} \times 10 \text{ mm} \times 2 \text{ mm}$  for a TiC deposit. To get the graphite substrates ready for the TiC coating, they were polished using progressively finer grit abrasive papers. Sample X was a graphite substrate polished with 400 grit TiC, Sample Y was polished with 1550 grit TiC, and Sample Z was polished with W0.5 emery grinding grease. Following polishing, acetone and methanol were used in an ultrasonic cleaner to clean the graphite substrates. Following the drying of the graphite substrates, CVD was used to provide a TiC coating on the substrate surface. During the deposition process for TiC coating, hydrogen served as the carrier gas to introduce methyltrichlorosilane (MTS, CH<sub>3</sub>SiCl<sub>3</sub>) into the furnace. The deposition rate of the TiC coating was also controlled by using argon as a diluent gas. The deposition furnace's temperature was regulated between 950 °C and 1050 °C during the deposition process. To achieve the intended coating thickness in this study, the deposition duration was regulated to 240 hours.

## Properties and its extents

In this research, the heat radiation of samples was measured in a direct fashion. As can be seen in fig. 1, an apparatus for measuring emissivity at high temperatures was employed. The main vacuum element of the system consisted of a turbomolecular pump connected in series with a mechanical pump. This set-up was established to achieve an extremely low air pressure (around  $10^{-4}$  mbar), with the aim of preventing any atmospheric turbulence. When samples were placed inside the inductance coil, they were heated by the induction furnace.



Figure 1. Diagrammatic representation of the heat radiation detection system

The mechanism for temperature monitoring was responsible for tracking the temperature levels of the sample. The infrared radiation signals emitted by the heated sample were detected using a Fourier transform infrared (FTIR) spectrometer equipped with a mercury cadmium telluride (MCT) detector. The samples' spectral emissivity was determined by comparing their heat radiation spectra to that of a blackbody standard [14, 15]:

$$\varepsilon(\lambda,T) = \frac{L_s(\lambda,T)}{L_b(\lambda,T)}$$
(1)

where  $L_s(\lambda, T)$  and  $L_b(\lambda, T)$  stood for the sample's and the blackbody's respective heat radiation intensities at a given wavelength,  $\lambda$ , and temperature, T. A straightforward computation of heat radiation spectrograms could yield  $L_s(\lambda, T)$ . Plank's Law provided the  $L_b(\lambda, T)$  calculation formula [16]:

$$L_b(\lambda, T) = \frac{C_1}{\lambda^5 \left[ \exp\left(\frac{C_2}{\lambda T}\right) - 1 \right]}$$
(2)

where the first radiation constant,  $C_1 = 3.742 \cdot 10^{-16} \text{ Wm}^2$ , and the second,  $C_2 = 1.439 \cdot 10^{-2} \text{ WK}$ , were measured.

## Evaluation of total emissivity

The ability to radiate heat over the studied wavelength range was characterised by the total emissivity, which was determined by integrating the emissivity across the spectrum. As per [17], the total emissivity was obtained by integrating the spectral emissivity  $\varepsilon_{\lambda}$  from  $\lambda_1$  to  $\lambda_2$ :

$$\varepsilon_T = \frac{\int_{\lambda_2}^{\lambda_2} \varepsilon_{\lambda} E_{b\lambda} d\lambda}{\int_{\lambda_2}^{\lambda_2} E_{b\lambda} d\lambda} = \frac{\sum_{\lambda_1}^{\lambda_2} \varepsilon_{\lambda} E_{b\lambda} \Delta\lambda}{\sum_{\lambda_2}^{\lambda_2} E_{b\lambda} \Delta\lambda}$$
(3)

where  $\lambda$  is the wavelength, T – the temperature, and  $E_{b\lambda}$  – the blackbody's heat radiation intensity.

Coating was identified using X-ray diffraction (XRD) analysis using Cu  $K\alpha$  radiation. The information was recorded digitally from 20° to 80° of angle (2 $\theta$ ).

## **Results and discussions**

The laser scanning confocal microscope measurements of the graphite substrate's surface roughness are shown in tab. 1. The surface roughness decreased from 3.01  $\mu$ m to 0.73  $\mu$ m between substrates X and Z. The cross-sectional shape of a TiC coating on a graphite substrate. The coating is compact and has a thickness of approximately 20  $\mu$ m. As a result, the coating qualifies as the perfect homogenous coating.

Substrate	Surface roughness [µm]	Polishing grit
Х	$2.96 \pm 0.13$	400#
Y	$1.28 \pm 0.11$	1550#
Z	$0.69\pm0.08$	W0.5#

Table 1. Roughness of the surface of graphite substrates

Simultaneously, the XRD data reveal that the coating's primary component is  $\beta$ -TiC, fig. 2. Sample Z has an interface that is quite smooth and free of defects. As thus, the substrate morphology dominated both the interface morphology and the interface bonding station. Ultimately, the heat transfer between the substrate and coating will be directly impacted by changes in the interface combination state.

Samples X, Y, and Z's spectral emissivity at 1050 °C and 1350 °C are displayed in fig. 3. The  $10^{-14}$  µm spectrum emissivity curvatures of all three specimen follow the classic *V-pattern* mode, with a dip in intensity followed by a rise. The spectral emissivity has a minimum value of 12.5 µm and a maximum value of around 10 µm. The spectral emissivities of samples X and Y differed significantly in this wavelength range, although Samples Y and Z's discrepancies were less pronounced. In accordance with the single Lorentz oscillator model, TiC's infrared emissivity exhibited unique variations in the  $10^{-14}$  µm wavelength range. The

reststrahlen zone of TiC is the name given to this unique region of spectral emissivity. The three samples' spectral emissivity displayed the TiC reststrahlen zone, indicating that the coating's heat radiation was mostly influenced by its material composition. Therefore, it could be concluded that the substrate was mostly responsible for the variations in spectral emissivity seen in the samples. At the same wavelength, Samples X and Z's spectral emissivity increased as their substrate surface roughness decreased. Additionally, the samples' spectral emissivity varied with wavelength, with values ranging from 8-12  $\mu$ m to 16-18  $\mu$ m.

10

12

14

1.0

0.9

0.8

0.7

0.6

0.5

0.4

0.3

0.2

6

Spectral emissivity



10

12

14

16

18

(a) Wavelength [μm]
 (b) Wavelength [μm]
 Figure 3. Three samples with distinct substrate morphologies were analysed for their spectral emissivity; (a) 1050 °C and (b) 1350 °C

16

18

Sample X's spectral emissivity varied very little with wavelength. Both samples Y and Z showed an increase in spectral emissivity with increasing wavelength in the aforementioned two wavebands, however Sample Z grew at a faster pace. As the temperature was increased from 1050-1350 °C, the spectral emissivity at the test wavelength increased in all samples. As temperature was raised, the gap between X and Y Samples expanded in terms of their spectrum emissivity. Three samples were prepared for TiC coating in this work using the same depositional conditions. As a result, it is possible to regard the coating's chemical composition, thickness, and surface shape as being identical across samples. The morphology of the substrate was the sole variable. Consequently, it is possible to draw the conclusion that the substrate shape significantly influenced the TiC coating's spectral emissivity. The spectral emissivity differential grew as the temperature rise, amplifying these effects.

0.3

0.2

Figure 4 displays the variation of total emissivity as a function of temperature, ranging from 1050-1650 °C, for the three materials under study. The diagram indicates that the average emissivity of the three samples rises at lower temperatures and subsequently declines at higher temperatures. The heat radiation characteristics, as described by infrared radiation theory, are primarily governed by factors such as carrier density, carrier mobility, and the frequency of carrier collisions [18]. As the measurement temperatures increased, the activity of carriers enhanced the electromagnetic energy that the materials were emitting. Materials with higher energy and the second se

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total emissivity of three samples

Consequently, as the coating's temperature rise, its heat radiation initially increased. However, as temperature climbed during measurements, the escalating interfacial stress caused the interface to separate. Due to defects at the interface, heat was reflected more and transferred less efficiently from the substrate to the coating, reducing the coating's radiative capacity. Ultimately, this reduction manifested as a lower overall emissivity of the sample at high temperatures.

Moving from Sample X to Sample Z, there was a reduction in substrate roughness, which correspondingly led to a decrease in the interfacial resistance to heat flow. This change facilitated a rise in the overall emissivity and resulted in Sample Y and Sample Z exhibiting similar patterns of variation in emissivity at the transition temperature. In line with the overall trend observed in the spectral emissivity, the total emissivity augmented from Sample X to Sample Z with the diminishing roughness of the substrate. When contrasted with Sample X, the emissivity growth rates for Samples Y and Z were notably higher, registering increases of 11.67% at 1050 °C for Sample Y and 29.65% for Sample Z, respectively. Increasing test tem-



Figure 5. Samples' overall emissivity in relation substrate roughness

gy emissions have better heat radiation capabilities. The failure to account for the substrate's effect on the heat radiation property is largely to responsible for this discrepancy. But in this investigation, the substrate was treated like any other part of the heat conduction system.

The structural configuration was envisioned with the substrate and the coating acting as layers in a composite structure. Given that the TiC coating and the graphite substrate expanded and contracted at dissimilar rates, stress occurred at their boundary. At the elevated temperatures in question, interfacial tension was considered minimal, allowing for efficient heat transfer from the substrate to the coating.

peratures were associated with a faster increase in total emissivity, indicating that substrate shape was more important than temperature in determining the heat radiation characteristics of TiC.

Figure 5 illustrates the relationship between emissivity at various temperatures and substrate roughness for the three different samples. The graph shows that the total emissivity of the samples decreases in an almost linear fashion with the increase in substrate roughness, regardless of the temperature. A model is presented to encapsulate this nearly linear relationship between the total emissivity and the substrate roughness [19]:

$$E = a \times x + b \tag{4}$$

In the given relationship, E represents the total emissivity, while a and b are constants that characterize the influence of surface roughness on emissivity, with x being the variable representing surface roughness. Here, a surface roughness, x, of 0 corresponds to the emissiv-

ity of the coating on an ideally smooth graphite substrate, as suggested by the intercept *b*. The constant *a* quantifies the effect that the substrate's roughness has on the coating's ability to emit heat radiation.

The constants a and b at various temperatures are determined using the relationship between surface roughness and total emissivity as outlined in eq. (4), with the findings. If the absolute value of a goes up, it means that the substrate roughness is having an increasingly detrimental effect on the heat radiation emitted by the TiC layer.

Figure 6 shows the *a* and *b* absolute values at the measured temperatures. The rising tendency continues until a temperature of roughly 1450 °C is achieved, after which the absolute values start to fall. As shown by the variation of, the shape of the substrate has a large impact on the TiC coating's heat radiation below 1450 °C but a less impact above this temperature. Similar to the parameter |a|, the value of *b* increased and decreased as temperature did. As previously indicated, *b* had to do with the coating's heat radiation on the perfect smooth surface of the graphite substrate. The variations of *a* and *b* with temperature showed that there was a strong temperature dependence for the substrate's influence on the coating.



Figure 6. (a) The results of a in absolute terms and (b) the variation of b with respect to temperature



Figure 7. The heat transfer schematic among the substrate and coating; (a) the smooth surface and (b) the rough surface

The heat radiation characteristic of a given coating is influenced by the properties of both the substrate it's applied to and the coating material itself. Figure 7 illustrates the coating diagram for two substrates that have either smooth or rough surface textures. For determining the apparent emissivity of a uniform coating within a layered structure:

$$\varepsilon(\lambda) = (1 - \rho) - \frac{\chi(1 - \rho)^2}{e^{2\alpha d} - \rho\chi}$$
(5)

where d is the coating thickness,  $\mu$  – the wavelength,  $\rho$  – the coating reflectivity,  $\chi$  – the substrate reflectivity, and a – the Lambert-Law Beer's absorption coefficient.

This formula showed a strong correlation between the coating's thickness, interface reflectivity, and coating reflectivity and its heat radiation property. The heat radiation from the substrate might pass through the coating if it was within a specific range. Thus, it was not possible to ignore the substrate's impact on the coating's heat radiation in this investigation.

The electromagnetic wave responsible for heat radiation experienced reflection, scattering, and refraction at the interface during transmission, as per the geometric optics principle. The heat radiation was dissipated as electromagnetic waves travelled from the substrate to the coating and interacted at the interface. In terms of power transfer, the contact area between the two materials can be compared to their thermal resistance. If the coating had been applied to a smooth surface, the potential for heat absorption by the coating could have been higher. A rougher interface between the substrate and the coating alters the heat transfer characteristics by increasing the surface area at the interface and the loss of energy in the heat flow. It implied that the coating's ability to radiate energy diminished as it reached thermal equilibrium, thereby lessening the coating's capacity to radiate heat. As a result, as substrate roughness increased, TiC coating's heat radiation property dropped.

#### Conclusion

The TiC coatings were placed on graphite substrates of varying morphologies for this study in order to determine how the substrate's form affects the coatings' heat radiation qualities. It was observed that both the spectral and overall emissivity of the TiC coatings were inversely related to the roughness of the substrate. Furthermore, the effect of the substrate morphologies on the coating's ability to radiate heat was more pronounced at temperatures below 1450 °C and diminished at temperatures above this threshold. Further investigation showed that the TiC coating's heat radiation qualities had degraded because of the rough interface. This roughness led to increased energy dissipation and more complex interactions with electromagnetic waves.

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