Functionally Graded Materials for Thermal Barrier Coatings in Advanced Gas Turbine Systems Research

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Functionally Graded Materials for Thermal Barrier Coatings
in Advanced Gas Turbine Systems
Biannual Report 8 - Final Report

1.0 Executive Summary

A combination of two new production methods, Reaction-Bonded Metal Oxide (RBMO) and electrochemical processing, have been utilized to create a functionally graded thermal barrier coating. Electrochemical processing, which includes both electrodeposition (EDEP) and electrophoretic deposition (EPD), has been used to deposit both the metallic and ceramic layers of the coating. EPD has been used to deposit the RBMO precursor powders, which exhibit the dual properties of both a metal and ceramic due to its composite nature. A summary of the FGM production methods and resulting characterization of the produced coatings for the eleventh and twelfth quarters (5/96 - 12/96), as well as a project summary, are outlined in this final report.

The RBMO process utilizes the oxidation reaction of attrition-milled and compacted metal/ceramic powder mixtures to make monolithic and composite ceramics. In the production of reaction-bonded Al_2O_3 (RBAO), the volume fraction of Al is usually in the range of 0.3-0.6, with the remainder of the powder mixture being Al_2O_3 and ZrO_2 , for example. The process offers significant advantages over conventional ceramic processing such as low raw material costs, low processing temperatures, machinability of green bodies, and near-net-shape forming capacity. Reaction-bonded ceramics/composites exhibit superior microstructures and fracture strengths; for instance $< 1 \mu\text{m}$ grain size and $> 800 \text{ Mpa}$ for the ZrO_2 -containing RBMO bodies.

The electrodeposition process has been characterized with respect to both the microstructure and resultant properties of electrodeposited Ni-Al- Al_2O_3 coatings. Multilayered

coatings of single and dual particle matrix composite have been produced by this process. The structure of the electrodeposited layers has been studied using both light and scanning electron microscopy. Variation in particle bath loading and current density has shown that the amount of alumina incorporated in a single layer can be controlled between a range of 0 to 40%.

Codeposition of Al has been shown to result in a refinement of the Ni matrix structure at high ($> 10 \text{ A/dm}^2$) current densities. For single particle baths, the codeposition of Al_2O_3 was more strongly affected by current density and bath particle content than was the codeposition of Al. However, for baths containing both Al and Al_2O_3 the amount of incorporated Al_2O_3 no longer depended on current density. With the choice of appropriate conditions, coatings of Ni with up to the previously mentioned limit of 40% Al_2O_3 were made. Similar experiments with Al yielded a maximum of 17.5 vol.% only. Uniform and graded mixed-particle coatings were also produced. When coatings containing Al were annealed, the reaction of the two elements resulted in the formation of either single phase γ or two phase γ - γ' alloys, in agreement with the equilibrium phase diagram. Mechanical properties of these layers have been evaluated and related to the microstructure through bend testing and microhardness measurements. Elevated-temperature testing of the electrodeposited layers has also been performed through an oxidation study and thermal cycling of the final thermal barrier coating (TBC). The oxidation studies have shown that multiple oxides tend to form; one that forms at the metal/gas interface and a second that grows through the electrodeposited layer. It was found that alumina addition to the nickel matrix did not result in an increase in oxide growth rates, and in some cases, lead to a decrease in the growth rate.

Electrophoretic deposition has been used to deposit the yttria-stabilized zirconia (YSZ) top-coat of the TBC, in addition to the RBAO layer. The deposition of YSZ is made possible by

the metallic nature of the RBMO layer. Electrophoretic deposition utilizes the process of electrophoresis, which is the migration of the charged particles toward the deposition electrode. The direction of the particle migration is controlled by the zeta potential of the particles. Final sintering of the YSZ and RBMO layers is done simultaneously, thus producing a fully ceramic top-coat. Microstructural characterization of both the as-deposited “green” state, and the sintered structure has been performed for both the RBAO and YSZ layers. The sintered microstructure shows a dense Al_2O_3 layer with corresponding microhardness values roughly an order of magnitude higher than that of the Ni matrix.

Characterization has been performed on the final electrochemically produced TBC. The results of both oxidation studies and thermal cycling will be discussed.

2.0 Research Progress and Results

2.1 Task B: Processing/Structure Characterization

2.1.1 RBMO

The RBMO process has been used to produce a dense, alumina-rich layer between the electrodeposited Ni-Al- Al_2O_3 layer and electrophoretically deposited YSZ layer. This process involves the electrophoretic deposition of the RBAO precursor powder, followed by two separate sintering runs. The first annealing step is a low-temperature (400°C) heating of the precursor-RBAO coated electrodeposited layers. This step is designed to encourage bonding between these layers, without significantly oxidizing the substrate or the RBAO powder. The second annealing step is the final sintering following the deposition of YSZ, which is a high-temperature process

(1050°C) The result is the formation of a dense fully ceramic layer consisting of both Al_2O_3 and ZrO_2 from the original RBAO precursor powder.

The RBAO powder used in this project is produced with an Al- Al_2O_3 - ZrO_2 ratio of 45:45:10 which becomes 20:70:10 following pre-deposition milling in ethanol.

2.1.2 Electrochemical Methods

Electrodeposition

The effect of particle concentration in the plating bath has been studied to determine the corresponding concentration in the coating. Figure 1a shows a plot of volume percent alumina in Ni- Al_2O_3 coatings as a function of volume percent in the bath for a range of current densities. It can be seen that increasing the amount of alumina in the bath resulted in a steep increase in vol.% alumina in the coating. A maximum of 40 vol.% alumina in Ni was achieved at 1 A/dm^2 for a bath loading of 5.3 vol.%. This value is almost twice the maximum reported by Ding et al. [1] for Ni electrodeposits that contained 2.7 μm α -alumina particles. The decrease in volume percent as a function of current density for our coatings followed a similar trend to that found by Ding et al. [1] for Ni- α alumina and Cu- α alumina and Celis et al. [2] for Cu- γ alumina deposits.

Compared with Al_2O_3 , the codeposition of Al was less strongly affected by current density and bath particle content. The amount of Al in the coating ranged from 5 to 17.5% only, as can be seen in Figure 1b. Comparison of the structure of the Ni matrix at high current densities ($> 10 \text{ A}/\text{dm}^2$) with and without the presence of Al showed that codeposition of Al resulted in refinement of the structure. Comparable studies in Ni- Al_2O_3 coatings were not possible, because the etchant preferentially attacked the interface [3]. However, from the variation of hardness of the coatings with particle vol.% we believe that at lower current densities, the alumina particles result in a

coarsening of the Ni grain structure. We have seen that the grain structure of Ni for sulfamate baths becomes finer with decreasing current density.[3]

In mixed particle baths, the codeposition of Al was not affected by the presence of alumina. However, the codeposition of alumina was suppressed at lower current densities so that the two lines shown in Figure 1a for low and high current density regimes collapsed onto the latter. The reason for this behavior, we believe, is related to the distortion of the field lines around metallic (conducting) versus ceramic (insulating) particles during deposition.

Graded coatings of Ni-Al-Al₂O₃ were produced by varying the bath particle content at a fixed current density of 5 A/dm². A light optical micrograph of one such coating is shown in Figure 2a and the annealed structure of the same coating in Figure 2b. When coatings containing Al were annealed, the reaction of the two elements resulted in the formation of either single phase γ solid or two phase γ - γ' , in agreement with the equilibrium phase diagram. The sample in Figure 2b, annealed for 1 hr at 635°C, was a two phase mixture of γ - γ' , with the alumina particles residing in the γ phase.

Figures 3a and 3b present the hardness of single-particle, uniform coatings of Ni-Al₂O₃ and Ni-Al as a function of current density. Two points are worth noting. First, at high current densities (> 10 A/dm²) the “soft” metallic Al particles resulted in greater hardening than the “hard” ceramic Al₂O₃ particles. Second, at lower current densities the incorporation of alumina resulted in a smaller increase in the hardness than at higher current densities, even though the volume percent of the incorporated particles was larger at the lower current densities. In other words, for both types of coatings the hardness did not follow a simple rule of mixtures. The reason for this, we believe, is the change in microstructure of the Ni matrix when the second phase particles are incorporated.

The elevated-temperature evaluation of the electrodeposited layers has also been performed. A series of experiments were conducted to determine the role of alumina with respect to the oxidation rate of the Ni matrix. Figures 4-6 show characteristic micrographs of oxidized single plates of nickel, nickel-alumina, and a discretely layered structure (up to 30 vol.% alumina), respectively. Two different types of oxide growth were observed. Type I oxide was seen to grow at the oxide/gas interface. This type of oxide growth is indicative of nickel ion diffusion outward (p-type). Type II oxide can be seen to grow within the coating. The growth of this type of oxide would indicate co-diffusion of the species; oxygen diffusion inward or the growth of a spinel phase in the nickel-alumina composites. The model system of nickel-alumina could be expected to form a spinel at temperature with the given partial pressure of oxygen within the furnace. The spinel, which forms according to the equation:

depends upon the co-diffusion of nickel and aluminum [4,5]. In Figure 6, the spinel growth can be observed around the alumina particles in the un-oxidized part of the coating. The particles that were initially agglomerated in the as-plated coating appear to have coalesced and grown. An energy dispersive spectrum (EDS) was obtained for both oxides, Figure 7 and 8. The only recognizable peak is nickel, suggesting that it is a nickel oxide (NiO). The absence of an aluminum peak does not suggest that the spinel did not form, but only that the system may be limited in detecting the relatively small amounts of Al present.

Figure 9 shows type I oxide thickness measurements as a function of time for various temperatures. With the exception of the nickel-15 vol.% alumina samples at 1000°C, the addition of alumina to the nickel matrix did not accelerate the growth of the oxide layer. Type II oxide was also measured and thickness measurements plotted in Figure 10. Again, it was observed that

growth of this oxide on the nickel-alumina electro-composites was not accelerated. Oxide formation at the surface of the electrodeposited layers was compared to oxide formation at the metal/ceramic interface of the final TBC. The results will be discussed later.

Electrophoretic Deposition

EPD has been utilized to deposit both the previously mentioned RBAO powder and YSZ (TOSOH, TZ-8Y). For both deposits, an electrical potential is created between the electrodeposited substrate and an aluminum anode. A photo of the EPD experimental setup can be seen in Figure 11.

RBAO is deposited from an ethanol suspension in a ratio of 30 g of RBAO powder to 180 mL of ethanol. The RBAO powder is milled for a minimum of 20 hours using sixty 1 cm zirconia balls as the milling media prior to each deposition. A potential of 100 V is applied for 3 seconds in order to create an RBAO layer approximately 25 μm thick. Figure 12 shows the characteristic grey color of this coating, in its as-deposited form, due to the high aluminum content. In contrast, Figure 13 shows the as annealed coating which is white indicating that the aluminum has oxidized, resulting in a 100% ceramic coating. The annealing process of the as-deposited RBAO is a two-step procedure. Following RBAO deposition, the coating is subjected to a low-temperature anneal to encourage bonding between the Ni-Al-Al₂O₃ electrodeposited layer and the RBAO layer. However, it is essential not to oxidize the RBAO during the process, or a fully ceramic outer layer will form and prevent subsequent YSZ deposition. To accomplish this, the sample is heat treated at 400°C for 10 minutes, with a heating rate of 3°C/min and cooling rate of 5°C/min. The slow heating and cooling rates are necessary due to the stresses that arise during the heating of a composite material.

Following the low-temperature annealing of the RBAO precursor, the YSZ top-coat layer is deposited. The bath composition for this system can be seen in Table 1. Using an applied voltage of 150V, a 15 second deposition will lead to a YSZ layer thickness of between 10 and 40 μm . This thickness is determinant upon the thickness of the preceding RBAO layer, which is less electrically conductive than the electrodeposited matrix. Therefore, thick regions of RBAO (corners and edges) tend to suppress deposition of subsequent ceramic layers.

A study was conducted to determine the relationship between applied voltage and the resulting deposition rate and green density of the produced coating. For this study, YSZ was deposited on a bare Ni substrate. Density measurements were made by measuring weight gain of the YSZ-deposited substrate using an analytical balance, and reading thickness contour measurements across the sample using a Tencor P-2 Profiler. Figure 14 shows a representative profilometer scan for one of the produced coatings. The uneven nature of the scan is due to the porosity of the as-deposited YSZ in addition to the roughness of the Ni substrate which is translated through the coating. Figure 15 shows a linear relationship between the applied voltage and corresponding deposition rate. Figure 16 shows a less clear relationship between deposition rate and green density. Due to the errors associated with this type of measurement, primarily dealing with the measurement of micron range thicknesses on an uneven substrate, no conclusions could be drawn from this data. However, we believe that rather than affect green density, deposition rate has a noticeable effect on sample surface characteristics. The samples with low deposition rates, corresponding to low voltage potentials, showed more microcracking and surface non-uniformities than samples deposited at high deposition rates ($> 150\text{V}$).

Figure 17 shows a scanning electron micrograph of the as-deposited interface between the RBAO and YSZ layers. It can be seen that the layers are intimately bonded, as the only visible

interface comes about due to the atomic number contrast between the respective metals in each layer (Al and Zr). Figure 18 shows a micrograph of the sintered interface, again showing good bonding between the two layers.

2.1.3 TBC Characterization

Figure 19 shows an optical micrograph of the outer 3 layers of a sintered electrochemically processed TBC. The interface between the electrodeposited and electrophoretically deposited layers is believed to be the area of potential coating failure. Therefore, several methods have been incorporated to fully characterize this region. Figure 20 shows a scanning electron micrograph similar to the optical image from Figure 19. The sample in both Fig. 19 and 20 was subjected to a 25 hour oxidation run, at 1000°C. In agreement with the previously discussed oxidation studies on the electrodeposited layers alone, the oxide regions which have formed have been identified as Ni-O, NiAl_2O_4 , and Al_2O_3 . The oxide layers were characterized qualitatively by a combination of microprobe analysis and energy dispersive spectroscopy. EDS was performed on the sample shown in Fig. 20, and each numbered point on this image has a corresponding EDS spectra. The region number, region identification, and corresponding EDS spectra number are listed in Table 2. The corresponding EDS spectra can be seen in Figures 21a-o. To assist in the identification of the phases in this region, the samples were studied in an electroprobe microanalyzer (EPMA). This probe map can be seen in Figure 22, showing the concentration of the elements Ni, Al, O, and Zr through the coating. Based on the above spectra, the oxide in the metal/ceramic interface region was identified. The NiO, NiAl_2O_4 , and Al_2O_3 regions are labeled in Figure 20.

In addition to the previously mentioned 25 hour oxide sample, four other TBCs were subjected to the same conditions for 5, 10, 25, 50, and 100 hours at 1000°C. The purpose of these experiments was to observe oxide growth at the metal/ceramic interface during extended periods at elevated temperatures. Figures 23a-f show this interface at the given times, in addition to one sample which was not oxidized beyond the final sintering step. There appears to be no growth in the oxide "fingers" which extend into the electrodeposited layer from the interface. It is believed that either aluminum is diffusing to the interface in the electrodeposited layer, or the RBAO layer is preventing the inwards diffusion of oxygen. Either or both of the processes prevent further formation of nickel oxide and spinel.

For additional high temperature evaluation of the coatings, thermal cycling has been performed by subjecting the samples to a temperature of 1050°C for 23 hours, followed by a rapid cooling and re-heating over the course of 1 hour. For comparison, 12 air-plasma sprayed commercial TBCs have been obtained from Westinghouse. These samples are sprayed on identical nickel substrates to the electrochemically deposited TBCs, with only the top and sides being coated. The Westinghouse samples consist of a MCrAlY bond coat and a YSZ top coat. Additional sample information from Westinghouse was not available due to the proprietary nature of these coatings. A total of 6 samples, 3 plasma sprayed and 3 electrochemically processed, were cycled for over 35 days. Following the 35th cycle, two samples were removed for analysis. The remaining 4 samples were cycled for a total of 55 days. Figure 24 shows a photograph of the latter 4 of these samples following cycling on the 35th day. Following this period, the majority of the original nickel substrate had oxidized and subsequently crumbled. The electrochemically processed samples remained intact, with the only visible oxide formation having occurred during the initial sintering run. One note should be made regarding the electrochemically processed

samples. The electrochemically processed samples all show some spallation around the edges of the sample. This is due to the fact that only the top face of the sample is coated. This allows for easy oxide formation at the edges, leading to the spallation seen in this picture. It is believed that a full coating of the sample would prevent this, effectively removing this pathway for oxygen to reach the critical metal/ceramic interface. Both sets of samples had begun to curl due to the stresses associated with coating of only one side. Again, this would likely be remedied by coating the entire sample.

Finally, to observe the thermally insulating effect of both the electrochemically processed and air-plasma sprayed samples, ΔT measurements were made through each sample. This testing was done by applying a heat source to the coating of each sample using an oxygen-acetylene flame. The flame temperature at the coating surface and corresponding bottom-side temperature was recorded for both type of TBC, in addition to uncoated nickel as a reference. Two samples were used for both type of TBC, on which multiple reading were taken over a range of temperatures. The surface and bottom-side temperatures, in addition to the corresponding ΔT values are shown in Table 3. Figure 25 shows this data graphically for the TBCs. From this data it can be seen that the electrodeposited and air-plasma spray samples performed similarly, and the TBC plays a major role in reducing the temperature to which the substrate is subjected. However, by normalizing the ΔT values based on thickness of each respective coating as shown in Figure 26, it can be seen that the electrochemically processed samples produced a greater per unit temperature reduction.

Conclusions

A graded thermal barrier coating has been produced by a combination of electrodeposition and electrophoretic deposition. This TBC has been shown to perform as well as a commercially produced air-plasma spray TBCs in an oxidizing, thermal cycling environment. Furthermore, the combination of the outer electrodeposited Ni-Al-Al₂O₃ layer and electrophoretically deposited and reaction bonded Al₂O₃ layer have shown to provide an excellent barrier to oxygen diffusion, thus protecting the substrate from oxidation.

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Table I

Bath composition of the electrophoretic bath (for 100 mL)

Acetone	26 mL
Methyl Iso-butyl Ketone	40 mL
Methanol	31.2 mL
Hexanol	2.8 mL
Nitrocellulose	0.1 wt%
Yttria Stabilized Zirconia	16.5 g

Table II

Identification of Oxides Present at Metal/Ceramic Interface

<u>EDS Number</u>	<u>Region Identification</u>
1	ZrO₂
2	ZrO₂
3	RBAO / NiO
4	RBAO / NiO
5	RBAO / NiO
6	RBAO / NiO / NiAl ₂ O ₄
7	RBAO / NiAl₂O₄ / Al ₂ O ₃
8	NiAl₂O₄ / Al ₂ O ₃
9	NiAl₂O₄ / Al ₂ O ₃
10	NiAl₂O₄ / Al ₂ O ₃
11	NiAl ₂ O ₄ / Al₂O₃
12	Ni / Al ₂ O ₃ (matrix)
13	NiAl₂O₄ / Al ₂ O ₃
14	NiO / NiAl ₂ O ₄

Note: **Boldface** indicates primary constituent.

Table III

Surface and Bottom-Side Temperature Measurements for TBCs and Uncoated Ni Substrates

<u>Sample</u>	<u>Thickness (μm)</u>	<u>Surface ($^{\circ}\text{C}$)</u>	<u>Bottom-Side ($^{\circ}\text{C}$)</u>	<u>ΔT ($^{\circ}\text{C}$)</u>
Air-Plasma Spray	401	1100	715	385
	388	1080	712	368
	401	1040	780	260
	401	1005	689	316
	401	985	705	280
	388	975	667	308
	401	950	681	269
	401	940	654	286
	388	893	624	269
EDEP	200	1060	736	324
	200	1056	737	319
	200	1017	680	337
	228	1005	677	328
	228	982	662	320
	200	975	673	302
	200	963	666	297
	200	930	629	301
	228	867	649	218
Ni		950	760	190
		940	740	200
		847	610	237

Figure 1: Volume percent (a) Al_2O_3 (b) Al in coatings as a function of particle volume percent in the bath for a series of current densities in the range of 1 - 25 A/dm^2 .

Figure 2: Light optical micrographs of a Ni-Al-Al₂O₃ coating. (a) From bottom to top, the three layers in the coating contain approximately 12-0, 9-16, 9-24 vol.% Al-vol.% Al₂O₃, respectively. (b) Same coating as in (a) annealed at 635°C for 1 hr.

Figure 3: Vickers hardness as a function of current density for (a) $\text{Ni-Al}_2\text{O}_3$ and (b) Ni-Al coatings. In both figures, the hardness of Ni with no particles is given for comparison.

Figure 4: Characteristic micrograph of a nickel electrodeposit after oxidation. Sample was deposited at 15 A/dm^2 , held for 10 hrs at 1000°C .

Figure 5: Characteristic micrograph of a nickel-alumina electro-composite after oxidation. Ni-30 vol% alumina, held for 10 hrs at 1000°C.

Figure 6: Characteristic micrograph of a stepped nickel and nickel-alumina electrodeposit after oxidation. Sample has a nickel inner layer with two composite layers containing 15 and 30 vol% alumina. Held for 10 hrs at 1000°C.

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Figure 7: Energy dispersive spectrum of type I oxide. Only a nickel peak is distinguishable.

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Figure 8: Energy dispersive spectrum of type II oxide. Again, only a nickel peak is discerned.

Figure 9: Oxide thickness measurements as a function of time for type I oxide at various temperatures: a) 600°C, b) 800°C, and c) 1000°C.

Figure 10: Oxide thickness measurements as a function of time for type II oxide at various temperatures: a) 600°C, b) 800°C, and c) 1000°C.

Figure 11: Photo of the electrophoretic deposition experimental setup. The cathode and anode are both nickel substrates and are oriented 1 cm apart parallel to each other. Voltage is supplied by a Kepco power supply. (not seen)

Figure 12: Reaction bonded coating deposited at 100 V for 5 sec, as deposited.

Figure 13: Reaction bonded coating deposited at 100 V for 5 sec, fired in air to 1050°C.

Figure 14: Profilometer trace showing the surface characteristics and thickness of a zirconia deposit on a nickel substrate. From this trace the coating thickness was estimated to be 43 μm . The surface roughness is a translation of the corresponding substrate roughness.

Figure 15: Plot showing the relationship between the applied voltage and deposition rate for the electrophoretic deposition of YSZ.

Figure 16: Plot showing the relationship between the deposition rate and corresponding green density of YSZ.

Figure 17: SEM micrograph of the as-deposited RBAO/YSZ interface. The RBAO layer was deposited for 4 seconds at a potential of 100V. The YSZ layer was deposited for 15 seconds at a potential of 150V.

Figure 18: SEM micrograph of the sintered RBAO/YSZ interface. Sample was sintered to 1100°C with heating and cooling rates of 1°C/min.

Figure 19: Light optical micrograph of the cross section of a four layered Ni-Al/Ni-Al-Al₂O₃/RBAO/YSZ coating heat treated to 1100°C for 3 hrs. The Ni-Al layer is not shown. The substrate is Ni.

Figure 20: SEM micrograph of the cross section of a four layered Ni-Al/Ni-Al- Al_2O_3 /RBAO/YSZ coating heat treated to 1100°C for 3 hrs. The Ni-Al layer is not shown. The substrate is Ni. The labeled points correspond to the EDS spectra given in Figure 21a-o.

Figure 21: Energy dispersive spectra corresponding to the points labeled in Figure 20. Spectrum a) shows peaks for the entire metal matrix/ceramic interface. Remaining spectra are numbered 1 through 14.

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Figure 22: JOEL 733 Super Probe spot map of an electrochemically processed TBC sample oxidized for 25 hours at 1000°C. Similar cross section to the image shown in Fig. 20. XVID image is a composite image showing all elements.

Figure 23: Light optical micrograph of the cross section of oxidation samples following various exposure periods. Samples were held at 1000°C for the following time periods: (a) sintered only, (b) 5 hours, (c) 10 hours, (d) 25 hours, (e) 50 hours, (f) 100 hours.

Figure 24: Light optical micrograph of thermal cycling samples following 35 cycles. Cycles consist of 23 hours at 1050°C with a 1 hour rapid cool to room temperature. Top two samples are air plasma sprayed samples prepared by Westinghouse. Bottom samples were electrochemically processed.

Figure 25: Graph showing the top side flame temperature and resulting reduction in temperature through the thermal barrier coating. Temperature reduction is normalized for each sample by dividing by the coating thickness.

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