

Investigation of New Additive Manufacturing DED Application for Waste-to-Hydrogen Conversion [†]

Svetlana Boshnakova

Center of Competence “Smart Mechatronic, Eco- and Energy-Saving Systems and Technologies”,
Technical University Sofia, Branch Plovdiv, 4000 Plovdiv, Bulgaria; boshnakova.sv@bswins.com

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Abstract

Relatively low-cost titanium carbide (TiC) materials and metal matrix composites (MMC) are proposed for waste-to-hydrogen conversion. Two types of steels are used as bases prepared from EN 10088 flat products, namely X2CrTi12 (1.4512, AISI 409) and X5CrNi18-10 (1.4301, AISI 304). TiC is mixed with TRIBALLOY[®] T-800 alloy in powder form and applied via laser-directed energy deposition (DED-LB) over the substrates. For the powder mixture, Fourier transform infrared spectroscopy (FT-IR) and differential scanning calorimetry (DSC) are performed. The raw materials are investigated for the processes that occur in them under heating. After the solidification of the molten mixture, grinding and polishing are performed to achieve a thin layer. The studies of the obtained MMC include interface zone assessment, hardness and Young's modulus distribution, microstructural analysis, and visual defect evaluation. Advanced sensors for acoustic emission (AE) and Electrical Contact Resistance (ECR) provided characterization together with micro-scratch testing. The use of photoluminescence spectroscopy is proposed for the new composite materials. The electron transfer pathway can be studied with time-resolved spectroscopy. Renewable energy production by breaking down waste into hydrogen-rich syngas can be achieved through pyrolysis, followed by steam reforming and purification. The obtained novel materials show promising application solutions with increased durability, corrosion, and wear resistance.

Keywords: additive manufacturing; directed energy deposition; composites

1. Introduction

Renewable energy production through the conversion of waste into hydrogen-rich syngas can be achieved via pyrolysis, followed by steam reforming and purification [1–8]. This paper proposes novel materials to facilitate the transition from carbon-based to green energy production by enabling the waste-to-hydrogen process, which involves three technical stages. First, the plastic/organic waste undergoes thermal decomposition in a rotary kiln via pyrolysis [2,4], producing biochar and high-calorific gas. Second, through reforming [1,3], the obtained gas reacts with superheated steam, converting complex hydrocarbons into syngas. Finally, the syngas undergoes purification, where water-gas shift [3,5] and pressure swing adsorption [2,7] reactions isolate high-purity hydrogen from the gas mixture.

The entire process requires advanced materials, particularly for the rotary kiln [4,7], where the primary pyrolysis occurs [2]. This study focuses on relatively low-cost titanium



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carbide metal matrix composites (MMCs) designed to ensure thermal stability and prevent corrosion during the decomposition of the waste [4].

The scientific novelty of this work focuses on the development and multi-modal characterization of TiC mixed with TRIBALLOY® T-800 alloy in powder form, applied via laser-directed energy deposition (DED-LB) over specific stainless steel substrates. The resulting novel coatings demonstrate promising application potential, offering increased durability, corrosion and wear resistance under the actual service conditions of high-temperature (500–900 °C) syngas atmospheres and severe waste particulate erosion.

2. Materials and Methods

Various MMCs [2] were fabricated using DED-LB, allowing for the additive manufacturing of near-net-shaped parts with variable surface geometries. A critical challenge for these new materials has been their application in the kiln's support system [4], specifically the bearings, which operate under extreme mechanical loads. These components rely on metal-to-metal contact; therefore, the new additive manufacturing (AM) materials must minimize friction and wear while ensuring structural integrity and smooth rotation under intense thermal loads.

Stainless steels were selected as raw materials for the first layer or base—X2CrTi12, a titanium-stabilized ferritic stainless steel designed for durability with a body-centered cubic crystalline structure and X5CrNi18-10—a very widely used austenitic stainless steel with high-chromium and nickel content, featuring a face-centered cubic lattice. These structural variants represented the most common crystal systems in industrial applications, making their properties significant for AM material integration. A mixture of TRIBALLOY® T-800 and TiC in powder form was applied over the stainless steel substrates. The particle size was between 45 and 150 µm (−100/+325 mesh), with a volumetric ratio of 3:1 between the two components [9]. The chemical composition of the base materials is provided in Table 1.

Table 1. Chemical composition of base metals (% content by weight).

% Content of Elements in Stainless Steels												
	C	Si	Mn	P	S	Cr	Ni	Ti	N	Mo	Cu	Fe
X2CrTi12	0.03	1.0	1.0	0.040	0.015	12.5	-	0.64	0.025	-	-	rest
X5CrNi18-10	0.07	1.0	2.0	0.045	0.015	18.50	9.00	-	-	-	-	rest

TRIBALLOY® T-800 is a cobalt–chromium–molybdenum alloy specifically designed for extreme wear and high-temperature oxidation resistance in harsh industrial environments [10]. Its chemical composition is shown in Table 2.

Table 2. Chemical contents of TRIBALLOY® T-800 (% content by weight).

% Content of Elements in Triballoy T-800									
Co	Cr	W	C	Ni	Mo	Fe	Si	Others	
Bal.	17.5	-	<0.08	<1.5	29	<1.5	3.7	<1.0	

The method of application was based on AM, which is increasingly adopted for its layer-by-layer production, enabling variable composition per layer. This approach can reduce work time and costs by up to 80% while minimizing material waste. The specific AM method was DED-LB. This technique allowed for the additive manufacturing of variable geometries, such as the cylindrically profiled bearing shells [2,4,10]. The method of directed

energy deposition by means of laser arc technology with powder feedstock enabled the molten metal to be added to build up the contact layer, which, due to the TiC on the surface, acted as microdimples that accommodate lubricating oil, thereby increasing the load-bearing capacity and reducing friction [10]. The equipment for laser metal deposition was a Rofin Sinar Nd:YAG 2 kW robotized system. The laser source was a neodymium-doped yttrium aluminum garnet laser with a wavelength of 1.06 μm . Parameters of the process were for each material specific. The lens setting was with a focal distance of 200 mm, and the laser focus spot was around 1 mm. Subsequently, a power of 1800 W was received, around 230 kW/cm^2 [11]. Using these parameters, samples with stainless steel bases and a TRIBALLOY® T-800/TiC layer thickness of 1–1.5 mm were obtained.

3. Results

Due to the novel nature of the TRIBALLOY® T-800 with TiC powder mixture, it was necessary to characterize both the powder and the final finished samples. The results of the X5CrNi18-10 with coating of TRIBALLOY® T-800 with TiC have been reported below.

3.1. Powder Characterization

3.1.1. Fourier-Transform Infrared Spectroscopy/FTIR

The FTIR spectra of the TRIBALLOY® T-800 with TiC powder mixture were acquired using a PerkinElmer Spectrum Two FTIR spectrometer (PerkinElmer Inc., Waltham, MA, USA), equipped with a universal attenuated total reflection accessory. The measurements were performed within the spectral range of 4000–400 cm^{-1} at a resolution of 4 cm^{-1} with 32 co-added scans. FTIR was a characterization method for the chemical structure of the powder used for the coating. By analyzing the infrared absorption, the functional groups and molecular bonds, essential for surface adhesion and durability, could be identified. This ensured that the coating material would achieve the precise chemical properties required for high-performance and exceptional wear and galling resistance [9,10]. Figure 1 shows the corresponding FTIR spectra of the main groups in TRIBALLOY® T-800 with TiC, observed at 3431.82 cm^{-1} , 2921.05 cm^{-1} , 2852.37 cm^{-1} , 1631.53 cm^{-1} , 1164.85 cm^{-1} , 1088.69 cm^{-1} , 797.98 cm^{-1} , 780.89 cm^{-1} , 695.25 cm^{-1} , 591.72 cm^{-1} , 516.38 cm^{-1} , and 458.53 cm^{-1} .



Figure 1. FTIR spectroscopy of TRIBALLOY® T-800 with TiC.

3.1.2. Thermogravimetry (TG), Differential Scanning Calorimetry (DSC) and Derivative Thermogravimetry (DTG)/TG-DSC-DTG/

Simultaneous thermal analysis (STA), incorporating TG, DSC and DTG (TG-DSC-DTG) was performed using a Netzsch STA 449 F3 Jupiter thermo-analyzer (NETZSCH-Gerätebau GmbH, Selb, Germany). DSC method was chosen in order to define the thermal properties of the powder [10,12]. By measuring the heat flow during phase transitions, critical parameters such as the melting point and the onset of thermal decomposition could be identified. This data was essential to ensure that the coating cured effectively and maintained its structural integrity under extreme operating temperatures. As illustrated in Figure 2, the thermal behavior and phase transformations of the analyzed sample were determined using STA from room temperature up to 1200 °C. The TG green line curve showed a continuous increase in the mass of the investigated powder starting above 600 °C, reaching a final residual mass of 102.66% at 1205.3 °C. Such an increase in the mass could be indicative of an exothermic oxidation reaction during these temperatures. The DSC profile, shown by the blue line, displayed several distinct thermal events corresponding to different phase changes and reactions. The one below 600 °C was endothermic, attributed to moisture desorption. Between 600 °C and 900 °C, multiple weak thermal fluctuations have been observed with corresponding peaks at 657.0 °C (−0.1354 mW/mg), 704.0 °C (−0.1136 mW/mg), 794.3 °C (−0.0074 mW/mg) and 865.1 °C (−0.0909 mW/mg). They could be interpreted as complex, multi-stage solid-state phase transformations or the onset of localized oxidation. Above 900 °C, it has been viewed as an exothermic reaction, characterized by major peaks at 1013.9 °C (0.1822 mW/mg), 1085.6 °C (0.3833 mW/mg) and a maximum peak at 1127.6 °C (0.5000 mW/mg). This signal directly correlated with the accelerated mass gain seen on the TG curve, confirming a major crystallization event. The DTG curve, with the purple dotted line, is relatively flat, indicating that the mass change rate has been slow and continuous rather than abrupt.

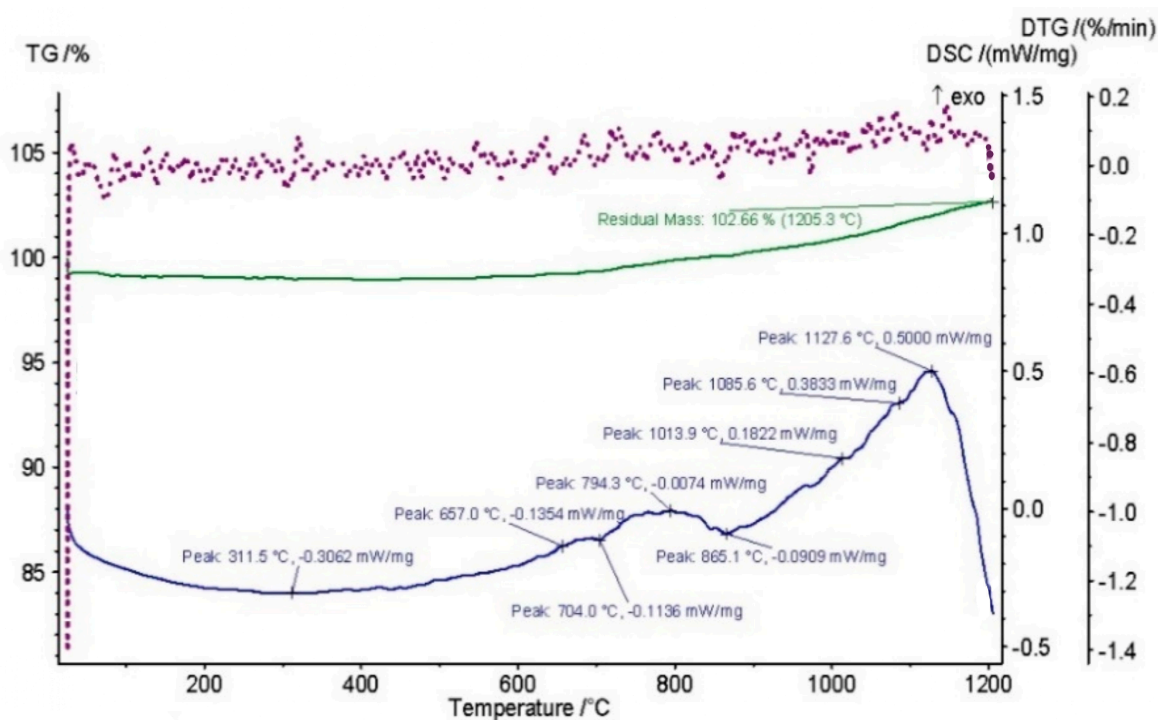


Figure 2. DSC analysis of TRIBALLOY® T-800 with TiC.

3.2. Post Application Characterization

After solidification of the molten mixture, grinding and polishing were performed. Visual examination revealed several surface faults; however, the resulting sample was deemed suitable for implementation in the rotary kiln for the waste-to-hydrogen process [2,4,10].

3.2.1. Hardness Measurement, Young's Modulus and Microstructure

To evaluate the mechanical performance of the coating, instrumented indentation testing was conducted using a UMT-2M universal mechanical tester (originally CETR, now Bruker Nano Surfaces Division, San Jose, CA, USA) system for micro-indentation testing. This system allowed for precisely measuring the material's hardness and Young's modulus by monitoring the load–displacement curves in real-time [13]. These parameters have been crucial for determining the coating's resistance to plastic deformation and the elastic behavior under high mechanical stress. The results obtained for the hardness measurement were $H_v = 2.356$ GPa and for Young's modulus $E = 28.889$ GPa. The instrumented indentation profiling revealed a prominent mechanical upgrade across the coating zones. The composite coating exhibited a peak hardness of $H = 9.03$ GPa and a measured Young's modulus of $E = 121.43$ GPa. This elevated hardness has been attributed to the rigid reinforcement network of the TiC particles distributed within the cobalt-based TRIBALLOY matrix. In comparison, the substrate regions displayed much lower hardness values ranging from 1.22 to 2.36 GPa. The localized drop in the substrate's indentation modulus $E = 28.889$ GPa has been a typical artifact caused by thin-film edge effect compliance. TiC particles had a hardness of $H = 31.98$ GPa. The achieved high hardness of over 9 GPa in the MMC coating layer provided a strong structural barrier against abrasive forces and particulate erosion expected during waste-to-hydrogen syngas processing. The mechanical behavior and indentation characteristics of the material were evaluated through a depth-sensing indentation test, as shown in Figure 3. The load–displacement curve was obtained after a loading, holding and unloading sequence [14] under a maximum applied force of 2 N. During the loading phase, the observed indentation depth increased continuously with the applied load, with a maximum penetration depth of 7.15 μm . Minor findings were observed along the loading slope (around 0.45 N and 1.6 N), which might be attributed to localized plastic deformation. Upon reaching the peak load of 2 N, a distinct horizontal dwell stage was maintained, during which the indenter penetrated deeper into the material from roughly 5.8 μm to 7.15 μm under constant load. This pronounced displacement highlighted significant time-dependent plastic deformation. The subsequent unloading curve exhibited a characteristic elastic recovery phase. The elastic–plastic recovery behavior allowed for the determination of the final residual depth after complete unloading, which was measured at approximately 4.5 μm . The large area enclosed by the loading and unloading curves indicated substantial energy dissipation through plastic deformation, confirming that the material possesses a combination of both high hardness and ductile-like energy absorption capacity [9,10]. According to the literature benchmarks, pure TRIBALLOY® T-800 coatings applied via DED-LB significantly reduce the sliding coefficient of friction by up to 42% [15]. These coatings have provided superior wear resistance compared to untreated AISI 304/409 stainless steels, reducing specific wear rates to around $2.06 \times 10^{-6} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$ [15].

To examine the coating's surface morphology and cross-sectional microstructure, the Axiovert 200MAT inverted metallurgical microscope (Carl Zeiss Light Microscope GmbH, Göttingen, Germany) was utilized. The coating's uniformity, porosity and the quality of the interfacial bond with the substrate were evaluated by this high-resolution optical analysis. These observations were vital to confirm that the microstructural characteristics align with the desired mechanical performance. Microstructure is presented in Figure 4.

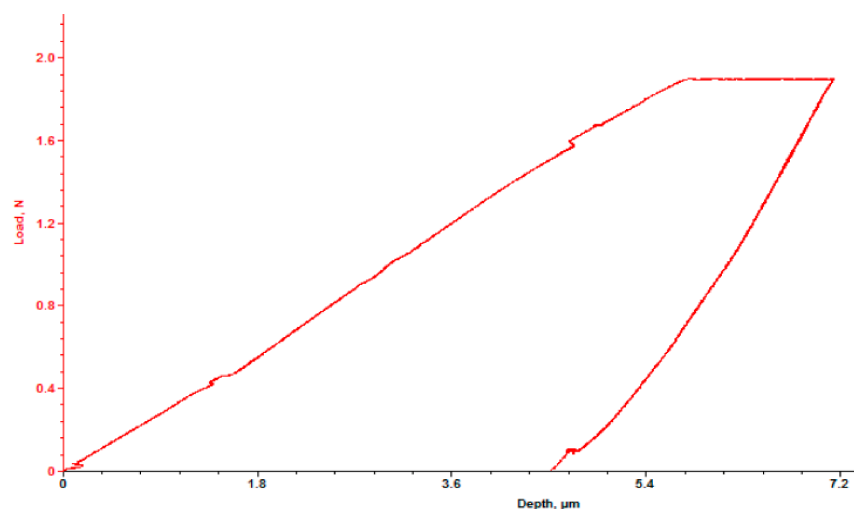


Figure 3. Result of the mechanical measurements load–displacement curve characterization of TRIBALLOY® T-800 with TiC.

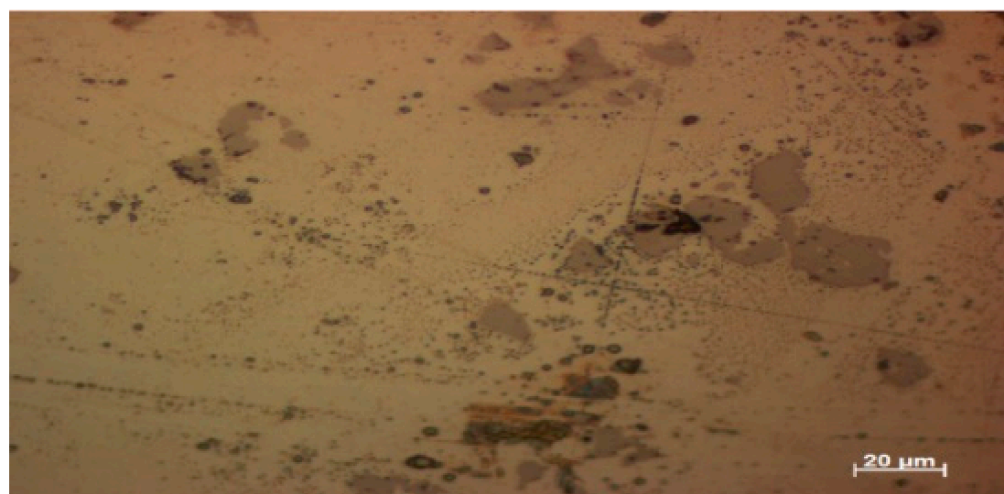


Figure 4. Results of the microstructure evaluation.

The microstructural analysis of the DED-LB-fabricated MMC coatings revealed a homogeneous distribution of TiC particles within the TRIBALLOY® T-800 matrix. Due to the high-energy density of the laser beam, partial dissolution of TiC occurred, leading to the precipitation of secondary carbide phases (such as $M_{23}C_6$ or M_6C) during rapid solidification. The matrix exhibited a fine dendritic and eutectic morphology, typical for DED-LB-processed T-800 alloy, which enhanced the overall hardness. No significant macro-segregation of TiC was observed, ensuring isotropic mechanical properties across the coating thickness. Porosity evaluation was conducted using image analysis software on cross-sectional micrographs. The overall porosity of the coatings on both X2CrTi12 and X5CrNi18-10 substrates was measured to be 1.35% and 1.20%, respectively. Isolated spherical gas pores were detected, primarily attributed to gas entrapment during the powder feeding process. However, the absence of interconnected porosity or crack networks confirmed the optimization of the laser parameters (power, scanning speed and powder feed rate). The interface zone between the MMC coating and both steel substrates demonstrated excellent metallurgical bonding, characterized by a distinct dilution zone without any delamination or cold laps. For the ferritic X2CrTi12 substrate, a narrow heat-affected zone was visible, while the austenitic X5CrNi18-10 substrate showed smooth epitaxial grain growth from the substrate into the coating. The AE and micro-scratch testing confirmed

high interfacial shear strength, as no acoustic emission signals corresponding to interfacial cracking or delamination were recorded prior to the plastic deformation threshold during scratching.

3.2.2. AE and ECR Scratch Test

Scratch testing was performed with the same UMT-2M universal mechanical tester because the system was equipped with in situ AE and ECR sensors. The tests were performed with a constant applied force of 6N along a 10 mm scratch path to analyze the internal and surface changes in the specimen. Acoustic emission functions by detecting elastic waves generated by micro-deformations and early-stage cracks within the material structure. By measuring ECR, it was possible to analyze the evolution of the contact surface and identify the formation of wear debris or any matter that has been crushed out. These methods provided a detailed diagnostic of the specimen's structural and surface degradation. The obtained shape of the AE signal was constant; some peaks attributed to the TiC particles were visible. It was concluded that no delamination or impermissible imperfections were found within the AM material. As observed in Figure 5 the readings of the AE sensor were constant between 0.01 and 0.02 V, with only a few peaks reaching 0.20 V. In this region, the TiC particles were detected using the acoustic wave. ECR sensors exhibited a continuous signal of 0.09 kΩ, showing no change in the electrical resistance, which confirmed the homogeneous nature of the coating.

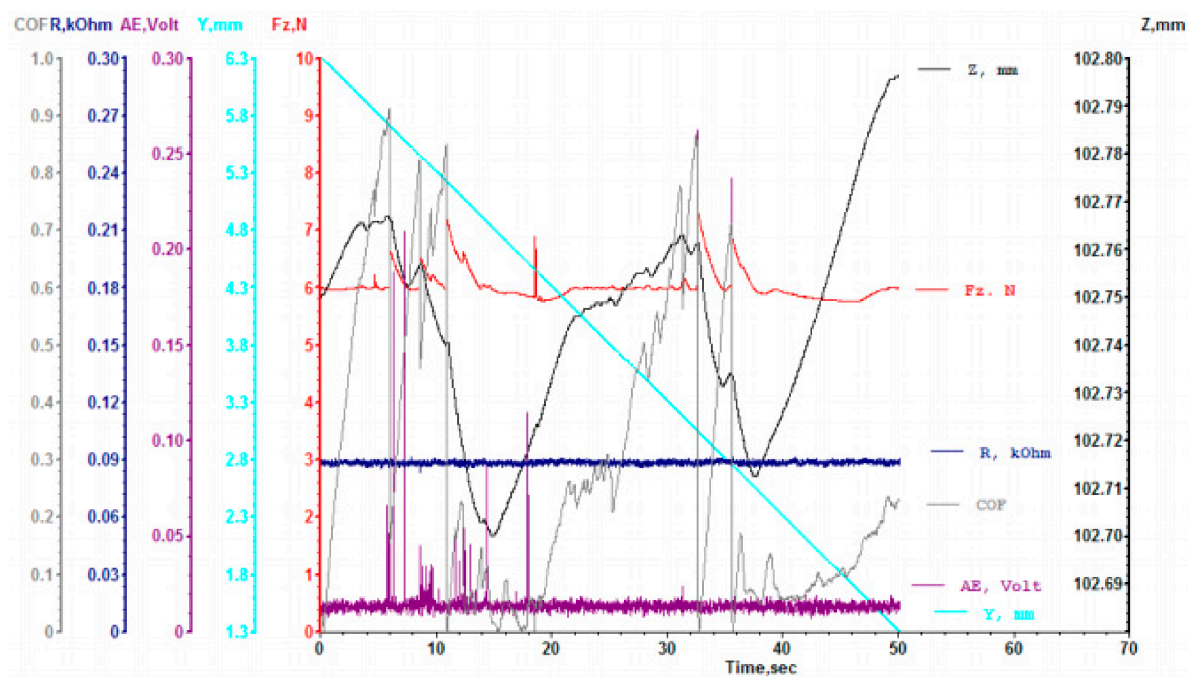


Figure 5. Results of the AE and ECR tests.

3.2.3. Photoluminescence Spectroscopy

Photoluminescence spectroscopy was utilized to investigate the electronic structure and optical properties of the material. By analyzing the light emission following laser excitation, point defects, impurities, and structural irregularities within the coating could be detected. This technique provided sensitive insights into the crystal quality and the presence of any radiative recombination centers that could affect the material's overall stability. Unfortunately, the AM material exhibited no photoluminescent response. In this study, photoluminescence was employed to investigate the electronic state and potential phase degradation within the laser-deposited matrix. Experimental observations revealed a

complete absence of photoluminescence emission across the investigated spectrum for the TRIBALLOY® T-800 with TiC composite. From a materials science perspective, this negative result could serve as a critical indicator of the coating's phase purity and structural integrity.

Both TiC and the cobalt-based TRIBALLOY® T-800 alloy are highly conductive metallic and intermetallic phases (such as Laves phase $\text{Co}_3\text{Mo}_2\text{Si}$ or CoMoSi), which inherently lack a semiconducting bandgap required to trigger radiative recombination or photoluminescence emission. Consequently, the absence of any detectable photoluminescence signal conclusively proved that no significant oxidation or formation of parasitic semiconducting oxide phases (such as TiO_2 or cobalt oxides) occurred during the high-energy DED-LB process. This confirmed that the protective atmosphere during laser processing was highly effective, preventing the degradation of the carbide reinforcement and ensuring a pristine, fully metallic/intermetallic composite structure. This high phase purity was directly correlated with the enhanced hardness and interfacial strength observed during the micro-scratch and acoustic emission testing.

4. Discussion

The mechanical performance of the investigated material correlated strongly with its thermal stability characterization. This relationship directly impacted the integrity and high-temperature behavior. The simultaneous TG-DSC analysis indicated a highly stable matrix below 600 °C, followed by a sequence of complex solid-state phase transformations at elevated temperatures. The multi-stage exothermic behavior observed in the DSC profile between 600 °C and 900 °C aligned with the onset of continuous mass gain, recorded on the TG curve. This collective thermal response suggested that the material underwent controlled phase restructuring involving the Laves phase of the TRIBALLOY® T-800 with TiC, which typically induced localized internal stresses. These microstructural characteristics directly influenced the room-temperature mechanical response, as quantified by the depth-sensing indentation measurements. Despite the high thermal stability and potential particle reinforcement (such as TiC or similar phases within the matrix), the material retained a substantial capacity for plastic relaxation under localized stress. Consequently, the material exhibited a well-balanced combination of structural rigidity, derived from its high-temperature phase evolution, and ductile-like energy absorption capacity, making it a viable candidate for demanding thermal and mechanical environments that can be found in waste-to-hydrogen applications.

5. Conclusions

The extensive characterization of the new AM material, composed of two layers—stainless steel and Tribaloy® T-800 with TiC—showed that it can be applied in the rotary kiln, achieving thermal and mechanical superiority. The use of FTIR, together with TG-DSC-DTG precise testing, unveiled the full behavioral spectrum of the novel powder. The hardness and Young's modulus measurements, microstructure evaluation, AE and ECR scratch tests and photoluminescence spectroscopy comprehensively mapped the AM material's properties. The interaction between the microstructure and mechanical properties shows durability and wear enhancement of the DED-LB-obtained surfaces. These findings support the potential of the composite coatings for extending the service life of critical components in waste-to-hydrogen conversion systems.

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Conflicts of Interest: The author declares no conflicts of interest.

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