



The Development of an Advanced Printable System for Metal/Ceramic Composite Materials

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ABSTRACT

Additive manufacturing is a process that combines the inherent benefits of ceramic and metal composites with the versatility of 3D printing. This approach is especially useful for creating ceramic and metal composites. Consequently, this makes it possible to create intricate structures that can be tailored to the specific requirements of the individual, and it also encourages the creation of innovative lightweight design solutions. The processing and fabrication of high-performance components are both processes that entail the use of this technology, which is now being utilized in a wide range of sectors, including the aerospace and automotive industries. With the intention of conducting this investigation, we designed a material extrusion 3D printing apparatus that has the potential to be used in the production of hybrid structures that are composed of ceramic and metal components. The method enables low-heat printing, which makes it feasible to debind printed samples in a straightforward and risk-free way by using a process that is based on alcohol. In turn, this not only enhances the efficiency with which high-performance composites are manufactured, but it also streamlines the flow of the process. Furthermore, in order to generate sophisticated structural components, this research included the procedure of creating composites of SUS316L/ZrO₂ that were printed using a 3D printer. This was done in order to fulfil the objectives of the study.

Keywords: Advanced Printable Material; Low-Heat Printing; Ceramic/Metal Composites.

1. INTRODUCTION AND LITERATURE REVIEW

Due to their exceptional high-temperature capabilities, wear resistance, and corrosion resistance, ceramics are considered a desirable material for various structural components (1,2). Several factors contribute to the appeal of ceramics for use in these components. Zirconia is a notable ceramic material recognized for its significant properties, including high stability at elevated temperatures, substantial bending strength and fracture toughness, as well as biocompatibility 3,4. Conversely, ceramics present challenges in handling due to their limited machinability and inherent brittleness. This limitation hinders the ability to construct large and complex structures, thereby reducing its overall utility. The material properties of ceramics present challenges in the working process. Consequently, there has been significant interest in the technique that facilitates the bonding of ceramics and metals. The process of linking metal and ceramics enables enhancements in material properties

that cannot be achieved by utilizing a single material alone. This creates opportunities for various potential applications. Our primary objective is to develop a 3D printing technique that can produce hybrid structures made from ceramics and metals. This constitutes one of our main objectives. In the fabrication of structural materials (6-7), oxygen sensors (9-11), and solid oxide fuel cells (SOFCs) (12-15), both metal and ceramic are essential components in the application of these materials. Metals and ceramics are commonly joined through welding, brazing, and diffusion bonding, which are established methods utilized for this purpose. There are several drawbacks associated with these methods, including the requirement for supplementary processes or materials to establish a connection. Consequently, there is a requirement for manufacturing techniques that do not necessitate any supplementary activities for connection purposes.

This study aimed to develop a dispenser-based metal 3D printing technology that operates at low heat and facilitates the debinding of the material through the application of the technology. A recently developed composite material is utilized, consisting of a blend of zirconia powder and stainless-steel powder, combined with a thermoplastic binder. This material serves as the medium for printing applications. The printed samples undergo debinding and sintering processes that are analogous to those employed in powder metallurgy. The debinding procedure can be conducted safely using alcohol, without the necessity for complex equipment. Utilizing the 3D printing technique we have developed, we aim to print hybrid structures composed of ceramic and stainless steel. Furthermore, the interface is examined through the application of the co-sintering process. Ceramics exhibiting corrosion resistance and high hardness can be integrated with metal components to produce composite materials. This procedure enables the integration of these ceramics with metal components. Additionally, components featuring three-dimensional ceramic structures are presented, demonstrating that the areas containing ceramics exhibit a greater degree of hardness compared to those without ceramics.

2. PRINTING SETUP

2.1 Material

The findings of this research study led to the development of a brand new material that was capable of being printed at a temperature that was far lower than the one that had been used before, which was about 80 degrees Celsius. Stearic acid, rapeseed oil, and a synthetic wax thermoplastic resin product known as PALVAX, which was created by Nippon Seiro Co., Ltd., were some of the other components that were used in the production process of the binder. After combining these components, they were heated in a microwave until the synthetic wax was completely dissolved in the rapeseed oil while being stirred to ensure that the mixture was well combined. It was necessary to accomplish this in order to complete the process of creating the binder. The gelatinization of the rapeseed oil was accomplished with the help of stearic acid. Stearic acid was the catalyst. Not only does stearic acid help to maintain the shape of the printed sample, but it also helps to ensure that the combined powders of ceramic and metal are distributed evenly throughout the sample. Stearic acid is a useful tool for both of these purposes.

Following that, the compound was formed by incorporating metal or ceramic powder into the binder, heating the mixture to 80 degrees Celsius in an oven, and stirring it using a planetary mixing equipment (SK-350T, Shashin Kagaku Co., Ltd.). This process was repeated until the compound was produced. This procedure was carried out several times until the compound was finalized. This method was carried out after the manufacturing

of the material after it had already been created. Powdered materials were used throughout the duration of this investigation. One of the powder materials that were used was stainless steel powder (SUS316L, manufactured by Daido Steel Group), which had an average particle size of around 10 μm . Additionally, zirconia powder (TZ-3Y-E, manufactured by Tosoh Corporation) was utilized, which had a particle size of 40 nm. In order to accomplish the objectives of their separate research, each of these powder compounds were used respectively. For the purpose of producing the binder, rapeseed oil, synthetic wax, and stearic acid were combined in a mass ratio of 89:9:2, respectively. A description was supplied before, and this was carried out in accordance with that description. During the process of printing, the powder and the binder were combined in a ratio of one volume to one volume. This was done in order to ensure that the printing process was successful.

2.2 3D Printing System

Acquiring data that delineates the print path through the segmentation of the 3D model into distinct layers is essential for executing 3D printing. We utilized PrusaSlicer, created by Prusa Research, a well-regarded slicing software for resin filament 3D printers, to achieve this objective. The study utilized the control software of the dispenser device, MuCAD (Musashi Engineering, Inc.), for its printing processes. The in-house Python application facilitated the conversion of G-code generated by the slicing software into the necessary code for operating the dispenser.

Figure 1(a) illustrates the 3D printing equipment utilized in this study. The 3D printing apparatus utilized was a dispensing device, specifically identified as the SHOTMASTER 300SX from Musashi Engineering, Inc. Shifting the stage in the vertical direction while simultaneously adjusting the dispenser along the horizontal and depth axes enables this printing method to produce a three-dimensional structure. Furthermore, the material underwent extrusion through an air pulse type dispenser (ML-5000XII, Musashi Engineering, Inc.), which facilitated the concurrent adjustment of geometry, movement speed, and compound extrusion pressure. Two ceramic heaters were affixed to the nozzle component, while a silicone rubber heater was applied to the syringe section, as heating is essential for the printing material utilized in this experiment. A digital fine thermometer (DG2N, Hakko Electric) was utilized to ensure a precise temperature of 80 $^{\circ}\text{C}$ was maintained. Prior to the printing process, the composite material underwent heating in an oven at 80 $^{\circ}\text{C}$ before being injected into the syringe. Subsequently, the syringe containing the substance was affixed to the printing device following a heating process to 80 $^{\circ}\text{C}$. The dispensing apparatus exerted pressure on the syringe's contents, resulting in their expulsion through a nozzle measuring 0.50 mm in diameter. An alumina board was secured to the stage for printing, and the compound was inscribed as the process unfolded in accordance with the established printing schedule.

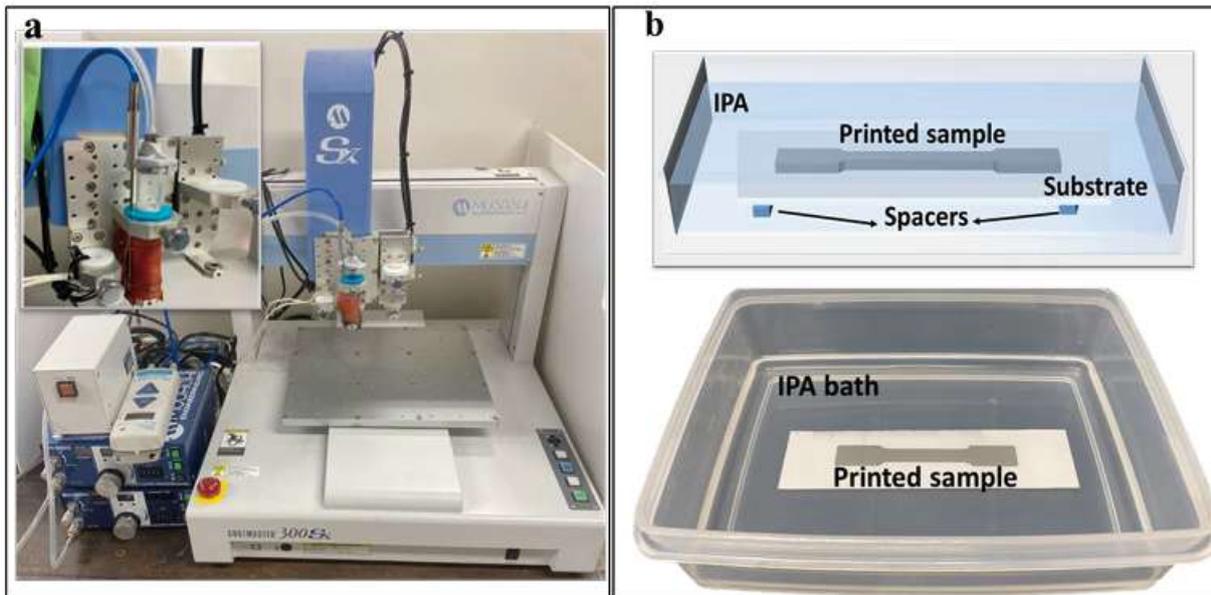


Fig. 1. (a) 3D printing system, (b) Degreasing system.

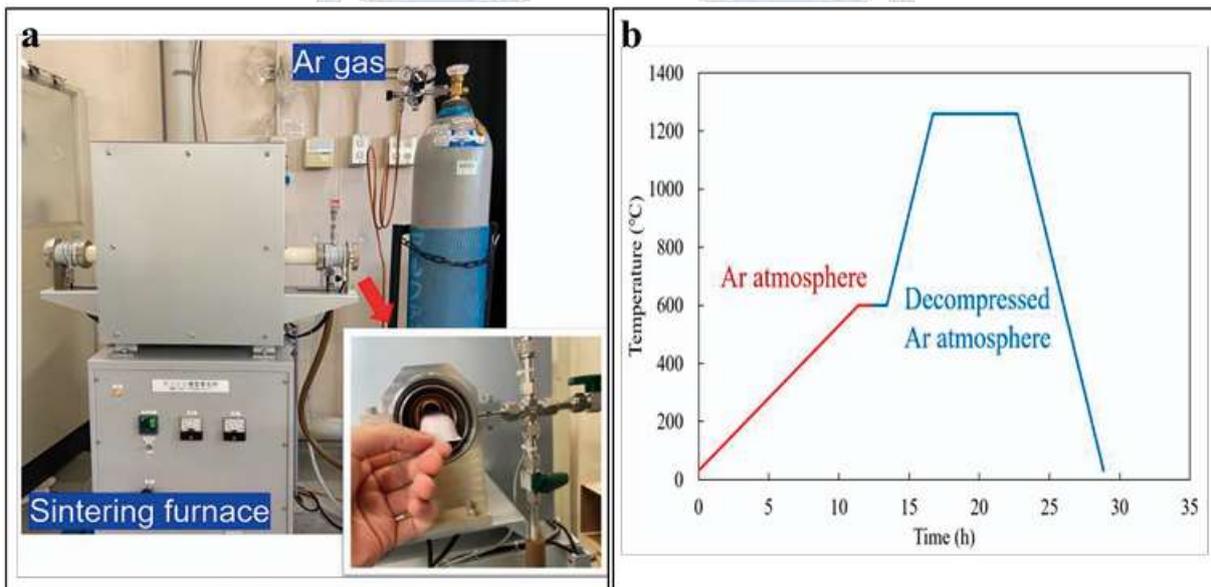


Fig.2. (a) Sintering system, (b) Variation of sintering temperature

2.3 Debinding and Sintering

The process of solvent debinding typically requires specialized equipment and the use of solvents that are classified as hazardous. Conversely, in this specific study, the synthesized binder can be readily debound by immersing the sample in an isopropyl alcohol (IPA) bath. This removes the necessity for the use of any specialized equipment. As depicted in Figure 1, the printed sample was maintained in the bath at room temperature for approximately 86 ks, which corresponds to one day. In this timeframe, the IPA undertook the task of dissolving the primary element of the binder, specifically rapeseed oil, followed by its removal. Conversely, the synthetic wax incorporated in the binder exhibited insolubility in IPA, enabling the printed sample to preserve its structure without sustaining any damage. Spacers were deliberately positioned beneath the alumina board supporting the sample to avert the accumulation of extracted rapeseed oil at the bottom of the IPA bath, as this could potentially obstruct the solvent debinding process. The sample that underwent debinding was placed in an electric furnace for the sintering process. In this experiment, the sintering procedure was conducted using a horizontal tube furnace equipped with molybdenum silicide heaters (HF-1500-S-T, Crystal Systems Co., Ltd.), as illustrated in Figure 2. (a). A cylinder of argon gas and a vacuum

pump were linked to the furnace to regulate the atmosphere during the sintering process. After the solvent degreasing process was completed, the samples were strategically placed in the center of the tubular furnace to facilitate the sintering process. Figure 2(b) illustrates the sintering process, detailing the temperature and ambient conditions that exist throughout this procedure. The essential criteria were established through a sequence of initial assessments conducted prior to the main analysis being performed. The initial phase involved heating the sample to 600 degrees Celsius at a rate of fifty degrees Celsius per hour in an argon atmosphere for a duration of 7.2 thousand seconds, or two hours. The residual binder present in the sample was eliminated throughout the entire operation through the process of heat breakdown. The adhesion between the metal and ceramic powders improved over this time period, resulting in the shape remaining unaltered even after the binder was removed. Subsequently, the environment was adjusted to a low-pressure argon atmosphere (spanning from 1 kPa to 100 kPa), and the specimen was heated to a temperature of 1350 °C at a rate of 200 °C/h. The temperature was sustained for a period of 21.6 ks, corresponding to six hours, to promote the bonding of the powder particles and improve the sample's density. Subsequently, the sample was gradually cooled to room temperature at a rate of 200 degrees Celsius per hour.

3. RESULTS AND DISCUSSION

Hardness and Tensile strength

A Vickers hardness instrument was used in order to ascertain the amount of hardness shown by the SUS and MIX samples, which included ZrO₂ volume fractions of 10%, 20%, 30%, and 40% correspondingly. The results of this determination were examined. The experiments were carried out with a load of 1.96 Newtons (0.2 kgf) and a loading time of thirty seconds throughout the whole of the procedure. As illustrated in Figure 3a, a cross-sectional representation of the SUS sample can be seen in this figure. To determine the level of hardness of the sample, 10 test locations were selected at random for the purpose of measurements. The diagram of the indentation is shown in Figure 3b as a consequence of the measurements of the hardness measurements. Figure 4 is a representation of the indentation diagrams that were generated after the completion of hardness testing on the surface of MIX samples that included varied percentages of zirconia according to the results of the tests. In the areas that were brilliant, zirconia was discovered, while SUS316L was discovered in the areas that were dark. The fact that the indentations are growing smaller and the diagonal lengths of the indentations are becoming shorter is a clear sign that the hardness is increasing greater as the dazzling sections get larger.

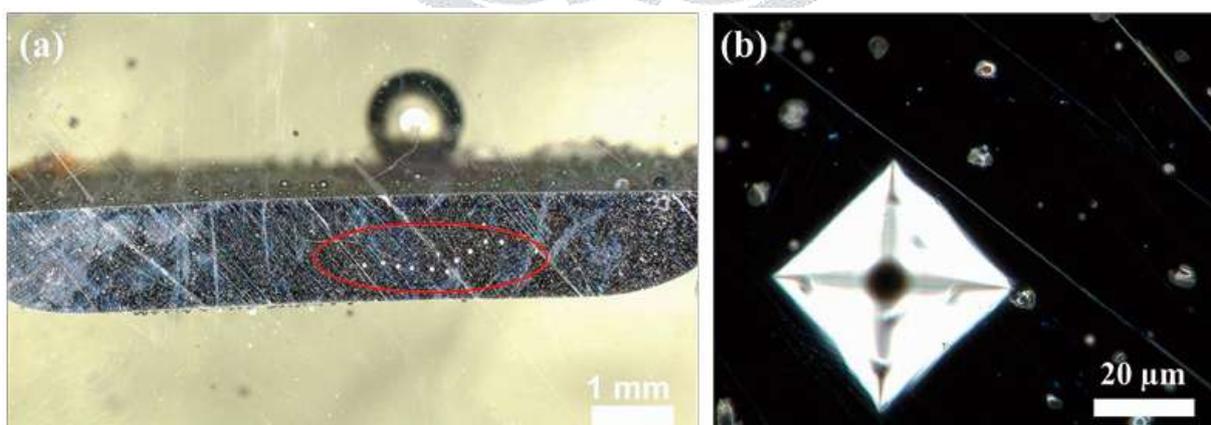


Fig.3. (a) Cross-section of the SUS316L sample; (b) Randomly selected indentations after hardness measurement.

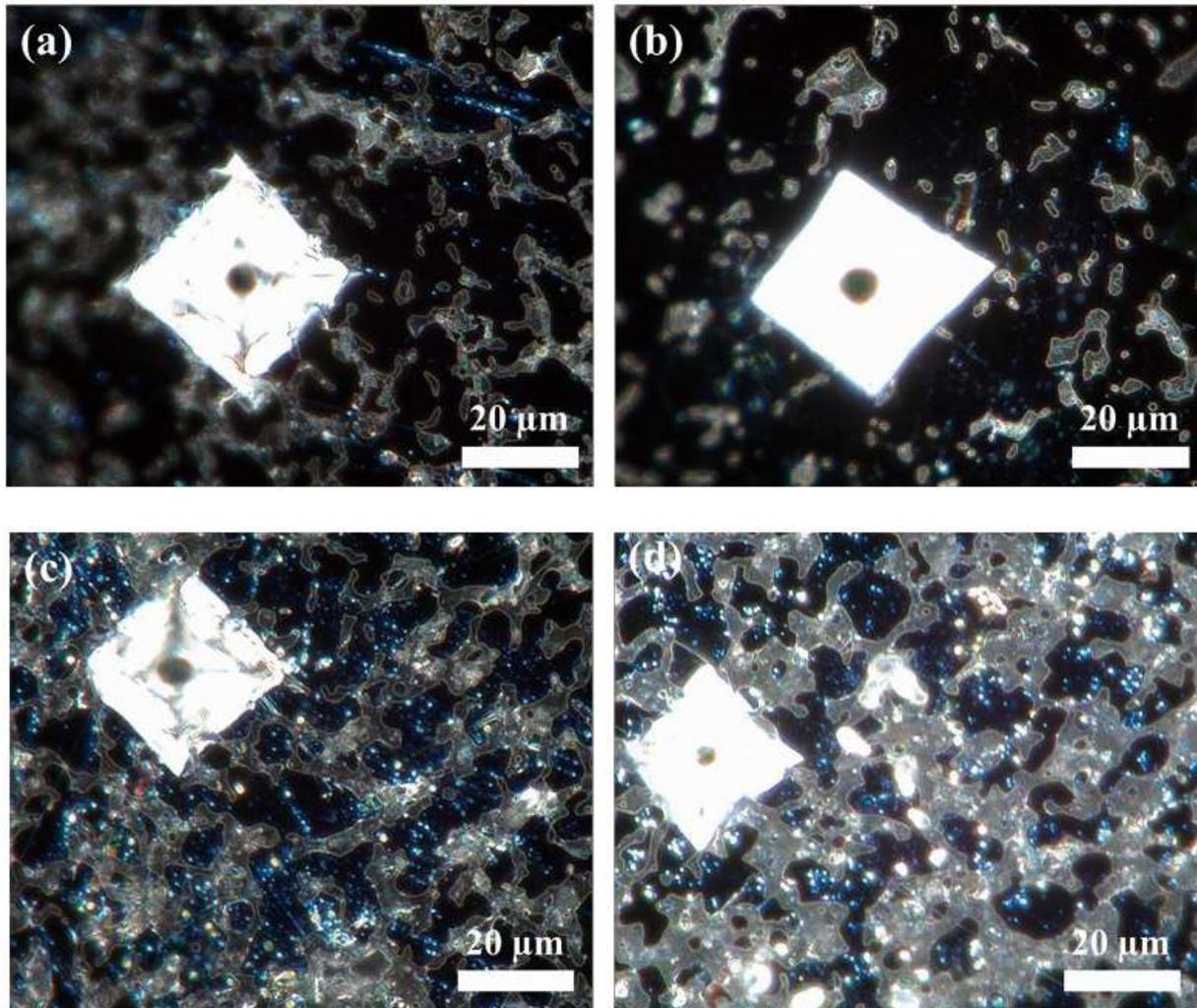


Fig.4. (a)-(d) Indentation plots of randomly selected surfaces of MIX samples with zirconia content.

Figure 5 contrasts the Vickers hardness values obtained for every sample. SUS: 123.5 HV, MIX (ZrO₂ 10 Vol%), 176.5 HV, MIX (ZrO₂ 20 Vol%), 203.4 HV, MIX (ZrO₂ 30 Vol%), 227.6 HV, MIX (ZrO₂ 40 Vol%), 350.3 HV. This suggests that the hybrid material MIX has more hardness than SUS316L, offering a new approach to get high hardness and wear-resistant zones on metal surfaces. The zirconia concentration of the MIX material employed in this work was regulated at 40 Vol%. This helps to prevent bending of the SUS-MIX composite and the visible cracking at the interface.

Nonetheless, zirconia's normal Vickers hardness is about 900 HV, hence raising the percentage of zirconia should help to further enhance the mechanical characteristics in the future.

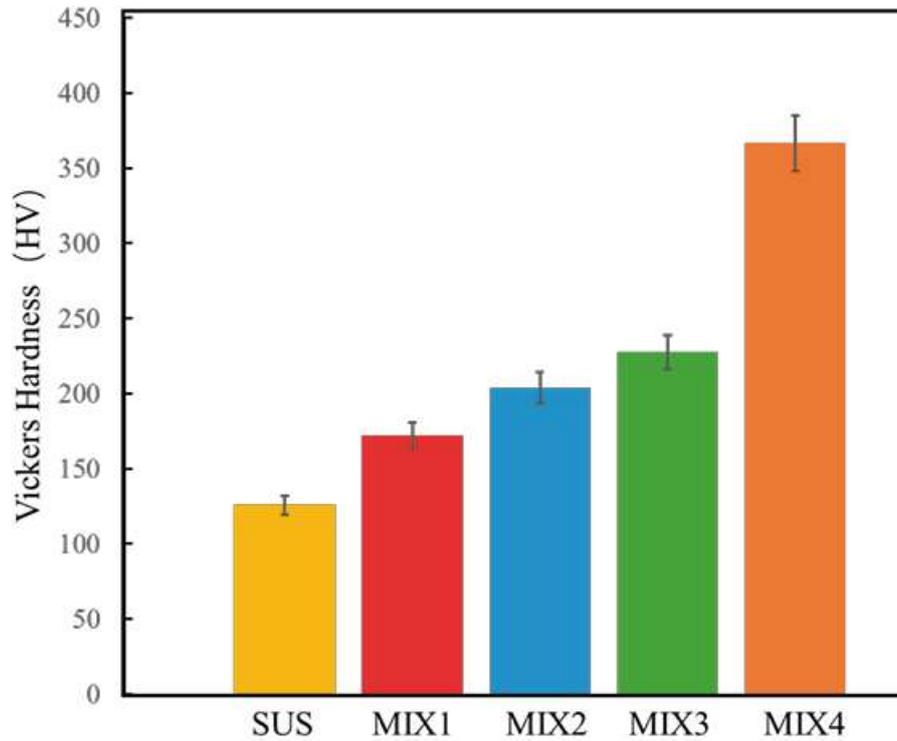


Fig. 5. Vickers hardness of SUS and MIX

4. CONCLUSION

A one-of-a-kind material has been found to be capable of being produced, which has the potential to enhance the process of low-temperature 3D printing and make the debinding procedure more straightforward to carry out. In addition to that, we developed a technique for the 3D printing of materials that utilizes this compound via the process of material extrusion. Our cutting-edge method allowed us to successfully create high-density samples via the process of co-sintering. This was accomplished by using our approach. The fabrication of hybrid structures employing zirconia powder and stainless steel powder was the method that was used to achieve this accomplishment. The novel chemical, which was made up of rapeseed oil, wax that was created, and stearic acid, demonstrated its efficacy in facilitating the debinding of solvents throughout the process by using an IPA bath. This bath was used to demonstrate the chemical's usefulness. Furthermore, we completed the successful building of a metal-ceramic hybrid structure by the amalgamation of stainless steel and zirconia with the compound, resulting in a sintered body that revealed no issues. The achievement of co-sintering metals and ceramics via the utilization of 3D printing is a substantial advancement, especially when one takes into account the inherent difficulties that are associated with this approach.

Furthermore, the significant hardness that is shown in the ceramic sections is suggestive of the potential for applications, such as the fabrication of mechanical components that have superior wear resistance on their surfaces. This is one example of the prospective uses. Therefore, this methodology provides a compelling way for the manufacture of hybrid structures that are formed of ceramics and metals when compared to other methods. The advantageous features that are intrinsic to both materials are shown here, along with the potential applications that may be made use of those properties.

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