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## A Statistical Prediction of Multiple Responses Using Overlaid Contour Plot on Hydroxyapatite Coated Magnesium via Cold Spray Deposition

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### Abstract

This work aimed to search optimal values for the properties of hydroxyapatite coated pure magnesium produced by cold spray deposition. Using fractional factorial design ( $2^{k-1}$ ), standoff distance, surface roughness and substrate heating temperature during cold spraying were selected as significant factors while number of sprays as insignificant factor. The responses chosen are coating thickness, hardness and elastic modulus of the coating. The analysis of the contour plots for thickness revealed that high thickness ( $>40\mu\text{m}$ ) was obtained when standoff distance is at 20-25mm, surface roughness at 240-700 grit and substrate heating temperature at 350-550°C. High nanohardness ( $>400\text{ MPa}$ ) obtained when standoff distance is at 20-35mm, surface roughness at 240-900 grit and substrate heating temperature at 350-550°C. High elastic modulus ( $>40\text{ GPa}$ ) was obtained at 20-30mm for standoff distance, 240-800 grit for surface roughness and 350-550°C for substrate heating temperature. The contour plot of coating thickness, nanohardness and elastic modulus were overlaid to find the feasible region.

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## 1. Introduction

Biomaterials are defined as any synthetic material that is used to replace or restore function to a body tissue and is continuously or intermittently in contact with body fluids<sup>1</sup>. The ideal biomaterials must meet a variety of different criteria, depending upon the application. The majority of orthopedic replacements are used in load-bearing situations such as total hip replacements or dental applications, resulting in the need for biomaterials with strong mechanical properties. In addition to providing mechanical strength the biomaterial must also be non-toxic to cells and living tissues<sup>2</sup>. Metal implants provide the most suitable mechanical properties for these applications, as polymeric and ceramic materials tend to be relatively weak or brittle. Titanium and titanium alloys, stainless steels, and cobalt-chromium alloys are all used in joint replacement procedures and generally provide suitable mechanical support to restore orthopedic function<sup>3</sup>. In comparison with titanium alloys and stainless steel, biodegradable magnesium alloys are identified as revolutionizing biometals. Magnesium (Mg) alloys have been suggested for biomedical application due to their potentials to serve as biodegradable metallic implants since they can be gradually dissolved, consumed or excreted in human body and then disappear after bone tissues heal<sup>4, 5</sup>. Magnesium ions present in the human body, whereby approximately 1 mol of Mg is stored in a 70 kg adult human body and an estimated amount of half of total physical Mg in the bone tissue. Elastic modulus of pure Mg is about 40-45 GPa which is very close to that human bone (10-40 GPa) so it can reduce the chance of stress shielding effects observed in the case of higher modulus materials such as titanium<sup>6, 7</sup>. In this study, pure Mg has been used as Mg alloys is generally not advisable because most alloying elements can be toxic to the human body.

The ability of metal implants to be incorporated into the natural bone structure, however, has posed problems and caused significant load-bearing differences that often result in implant failure<sup>8</sup>. To counteract this problem, various coatings have been applied to metal implants to promote bone growth and improve the implant-to-bone transition. A number of calcium phosphates have been shown to produce no adverse biological reactions when implanted within the body<sup>9</sup>. Furthermore, studies have characterized certain calcium phosphates, most notably hydroxyapatite ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ ), shown that bone adheres well to, and forms a bond with, these surfaces<sup>10</sup>. By coating metallic implants with calcium phosphates, it is possible to combine the mechanical strength of the metal with the biological compact of the mineral<sup>11</sup>. Calcium phosphate coatings have been shown to be successful in promoting bone apposition and growth in vivo<sup>12</sup>, and clinical studies have confirmed the efficacy of orthopedic implants coated with hydroxyapatite<sup>13</sup>. This success has spurred a significant amount of research aimed at understanding and optimizing the properties and behavior of these coatings. Conventionally, bioceramic such as HAP have been deposited by plasma spray technique. However, due to the inherent high temperature in plasma spray, the deleterious effect such as phase alteration, evaporation and debonding occur by this coating technique<sup>14</sup>. In this regard, cold spray can be a good alternative for coating deposition at temperature well below the melting point. Cold spray is a process in which solid powders are accelerated in a de Laval nozzle toward a substrate. Cold spray has emerged as a promising process to deposit nanostructured materials without significantly altering their microstructure whereas many traditional consolidation processes do<sup>15</sup>. In conventional cold spray technique hot gases at temperatures 500-700°C is used as carriers of the sprayed powders while the substrate remains at room temperature. In the present study, the cold spray technique was modified by using ambient air at room temperature as the spraying medium and heating the substrate to 400°C. This modification helped in retaining the HAP properties which usually show phase changes at high temperature deposition.

Design of experiment (DOE) is one widely used experimental study methods on many processes in engineering. It is a statistical approach in which a mathematical model is developed through experimental runs. Besides, it provides the researchers or users the opportunity to optimize and predict possible output based on the parameter setting<sup>16</sup>. In practice, the DOE method has been used successfully in several industrial applications for optimizing manufacturing processes. For example, DOE has been applied to optimize the plasma spray process of yttria-stabilized zirconia coatings<sup>17</sup>. Of the available DOE methods, a fractional factorial design is a variation of the basic factorial design in which only a subset of the run is used. These fractional factorial designs are among the most widely used types of

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