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# Nanostructure formation and its effects on the mechanical properties of kinetic sprayed titanium coating

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#### ABSTRACT

The nanostructure formation during kinetic spraying of commercially pure titanium (CP-Ti) were studied using transmission electron microscopy (TEM) and finite element modeling (FEM) considering conductive heat transfer. The high-velocity impacted particles, subjected to severe plastic deformation (SPD), were found to be tightly bonded, and also considerably homogeneous and randomly orientated equiaxed nanograins, including some recovered grains having a low dislocation density, were found to be formed over wide areas inside the coating due to strain accumulation and the resultant thermal history enhanced by subsequent impacts of the particles. The bimodal grain structure consisting of both larger grains having high-density dislocations (>250 nm) and smaller dislocation-free grains with non-equilibrium grain boundaries (<100 nm) was determined to be associated with both the strain hardening and the ductile dimple fracture of the coating.

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#### 1. Introduction

Kinetic or cold spraying is an innovative deposition process based on metallic or metallurgical bonding and mechanical interlocking of high-velocity impacted and severely deformed particles which experience strain-induced adiabatic heating, accompanied by shear instability [1–3]. Due to the low processing temperature and high deposition rate, this process is suitable for rapid production (e.g. spray forming) of oxidation sensitive materials, such as titanium [4].

The production of ideal bulk nanostructured materials (NSM) is of importance and is a challenge to materials scientists from both scientific and technological points of view because NSM possess the special and advanced properties (e.g. high strength combined with good ductility, high fatigue life, and superplasticity at relatively low temperature) [5-11].

It has been widely reported that an ultrafine grained (UFG) or nanocrystalline (NC) structure (~50–500 nm) has been formed in heavily cold-worked CP-Ti by SPD processes, including equal channel angular pressing (ECAP) [12,13], high pressure torsion (HPT) [14–16], ECAP and HPT [17], ECAP and cold rolling (CR) [18–21], hydrostatic extrusion (HE) [22], and surface mechanical attrition treatment (SMAT) [23,24].

In kinetic or cold spraying, which is also a SPD process, by utilizing TEM, it has been reported that an NC structure ( $\sim$ 20–50 nm) was formed in Al [25,26] and Ni [27] coatings with cryomilled (ballmilled under liquid nitrogen) powders, while an NC ( $\sim$ 30–50 nm) or UFG ( $\sim$ 100–250 nm) structure was observed only in the vicinity of the impacting interface of Cu [28–31], Al [32,33], Ni [29,34,35], and 316L austenitic steel [36] coatings without cryomilling of the powders. It has also been discovered that nanocrystallization from an amorphous phase in Cu-based bulk metallic glass (BMG) coatings occurred due to the large imposed shear strain at the interface upon impact [37].

However, reports of the deformation microstructures at the microscopic level of kinetic sprayed CP-Ti coatings are currently lacking in the literature, although some macroscopic aspects of bonding, deposition characteristics, and mechanical behaviors of the coatings have already been reported [4,38]. On the other hand, it was shown by Kim et al. [39,40] that an NC structure ( $\sim\!20\text{--}40\text{ nm}$ ) could be formed over the entire region of a single deposited CP-Ti particle ( $\sim\!5\,\mu\text{m}$ ) by warm spraying, due to thermally activated microstructural recovery processes (i.e. dynamic recovery and recrystallization) [41,42]. Moreover, from recent TEM investigations of two subsequently deposited CP-Ti particles by warm spraying [43], it appears that nanostructuring of the previously deposited particle may be enhanced. Nevertheless, detailed observations and analyses of the deformation microstructures in the coatings have not been reported.

Kim et al. [40] suggested that the formation of a fully NC CP-Ti coating would be possible by warm spraying or cold spraying

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with powder preheating. However, the issue of how the strain and temperature generated during spraying affect the nanostructuring of the CP-Ti coating needs to be addressed. To answer this question, we initially investigated the deformation microstructures in a dense CP-Ti coating produced by kinetic spraying without powder preheating [4] by using TEM. As a result, the gradual development of the nanostructure in a highly strained region (i.e. shear deformation zone) of the coating appeared to be influenced by strain, as well as by temperature, during the spraying process. In particular, the formation of considerably homogeneous and randomly orientated equiaxed nanograins with some dislocation-free nanograins (<100 nm) in the coating apparently seemed to be of importance because it most probably contributed to both the increased microhardness and ductile dimple fractures of the coatings shown in our earlier results [4].

Numerical modeling of deformation and interaction (i.e. fluid flow, heat transfer, and rapid solidification) of multiple molten droplets (such as titanium and tantalum) during thermal spraying has previously been performed [44,45]. In this study, numerical predictions from FEM computer simulations (considering thermal conduction) developed from a conventional model [4] will provide better understanding of the formation and evolution of nanostructure in kinetic sprayed CP-Ti.

#### 2. Computer simulation

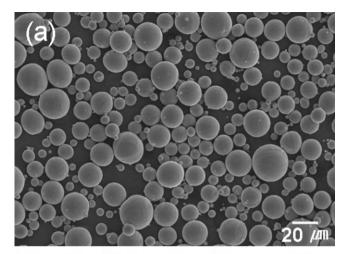
In order to predict and analyze the strain and thermal history of the deposited particles, non-linear transient FEM of the high-velocity micron-sized multiple particle impact process was performed using the commercial explicit code ABAQUS/Explicit [46]. Also, the Johnson–Cook plasticity model [1–4] was employed. The detailed model, boundary conditions, contact properties, and general/high strain rate material properties have been previously described [3,4]. Note that the particle impact velocities were assumed to be 800 m s<sup>-1</sup>, which is somewhat less than the velocity estimated empirically in our process  $(950 \,\mathrm{m \, s^{-1}})$  [4], to avoid abnormal termination of the simulation due to severe distortion errors of the elements during subsequent impacts of two dozen CP-Ti particles onto a mild steel substrate. Despite this assumption, one would expect reasonable predictions of the thermomechanical responses of severely deformed particles, which are probably comparable to the experimental results.

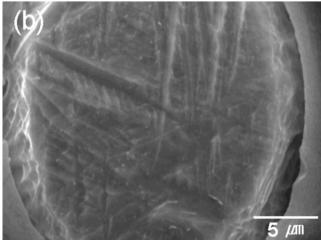
To investigate the effect of heat conduction on the thermal history of the deformed particles, fully coupled thermal stress analysis (i.e. a dynamic explicit procedure including thermal displacement), considering inelastic energy dissipation as a heat source (adiabatic heating) [2,46], was adopted, and conductive heat transfer based on the contact pressure transmitted across the interface [46] was specified for all contact surfaces.

The thermal conductivities of the heavily deformed materials were assumed to be 60% of those of the annealed bulk materials ( $\sim$ 17 W m<sup>-1</sup> K<sup>-1</sup> and  $\sim$ 52 W m<sup>-1</sup> K<sup>-1</sup> for CP-Ti and mild steel, respectively [47]), as suggested by Schmidt et al. [2]. During the total simulation time (10  $\mu$ s), the amounts of thermal convection and radiation were negligible, as compared to thermal conduction, and were not considered.

#### 3. Experimental

CP-Ti powder (mean size  $\sim 22~\mu m)$  was chosen as the feedstock (Fig. 1a). Detailed descriptions of the feedstock and spray coating methods (using helium as the process gas) were previously provided [4]. To compare the initial microstructure of the powder with the deformed microstructures of the as-sprayed coating, the coldmounted powder samples were mechanically polished (up to a





**Fig. 1.** (a) SEM surface morphology and (b) cross-sectional polished and etched micrograph of the powder.

 $0.3 \, \mu m$  alumina finish) and subsequently etched using a solution of 3 ml HF, 30 ml HNO<sub>3</sub>, and 67 ml distilled water. The cross-sectional microstructure of the powder and surface morphologies of the coating were observed using a field emission scanning electron microscope (JSM-6330F, JEOL). As can be seen in Fig. 1b, the cross-sectional polished and etched microstructure of the powder had an acicular martensitic structure. This peculiar structure, which resembles the structure of  $\alpha$ -titanium formed by laser melting [48,49], is mostly attributed to rapid solidification after plasma atomization of the powder which eventually made it difficult to identify each grain in the powder. Note that the microstructure of CP-Ti formed by plasma atomization is metastable but can be subsequently transformed into a stable form by post-heat treatment [48].

As seen in Fig. 2, the highly compacted particles appeared to be well-bonded, indicating that the particle impact velocity was sufficiently higher than the critical velocity required for successful bonding [1–3], as previously reported by Bae et al. [4]. The plane-view thin foils inside the coating (close to the substrate) were prepared by mechanically polishing the sample (up to  $\sim 30~\mu$ m), followed by electro-polishing using a twin-jet technique in a solution of 590 ml CH<sub>3</sub>OH, 350 ml C<sub>6</sub>H<sub>14</sub>O<sub>2</sub>, and 60 ml HClO<sub>4</sub> at a voltage of  $\sim 25~V$  and a temperature of  $-30^{\circ}$ C. The TEM investigations were conducted using a field emission transmission electron microscope (FE-TEM) (JEM-2100F, JEOL) operating at 200 kV.

The grain sizes of the deformed particles were estimated by measuring the mean diameters of the grains in the bright field (BF) and dark field (DF) TEM images using an image analyzer (Image Pro-

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