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# Characteristics of kinetic sprayed Ta in terms of the deposition behavior, microstructural evolution and mechanical properties: Effect of strain-rate-dependent response of Ta at high temperature



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#### ABSTRACT

Numerous efforts have been focused on the fundamental research regarding kinetic spraying processes, but previous studies were biased to only few kinds of metallic materials. In particular, for Ta with body-centered cubic crystal structure, systematic studies are currently lacking in the literature. In this study, the characteristic deposition behaviors, microstructures, and mechanical properties of kinetic sprayed Ta under various impact conditions were systematically investigated. Generally, unusual features were usually related to strain-rate-dependent response (i.e. Peierls-Nabarro short range barriers) of Ta at high-temperature. This made thermal softening effect to be saturated above a certain degree of temperature, contributing to inferior deposition efficiency for the pre-heating at 600 °C than those of 400 and 500 °C by creating synergy with powder oxidation. In terms of microstructural evolutions, the dislocation density was abnormally increased at a certain powder preheating temperature (i.e., 600 °C), owing to the strain-rate-dependent response of Ta at high-temperature. On the other hand, grain refinement generated by rotational dynamic recrystallization was mainly observed under the higher kinetic energy condition (He carrier gas) at RT. Especially, the dislocation density and volumeaverage domain size evaluated via high-resolution X-ray diffraction peak profile analysis were corresponding to the results of the microstructural observations. Additionally, the nanomechanical properties (i.e., nanohardness and elastic modulus) evaluated via nanoindentation tests were in good agreement with the results from transmission electron microscopy investigations.

#### 1. Introduction

A kinetic spray process (or cold gas dynamic spray process) is a solid-state bonding process in which feedstock powder is accelerated and deposited onto a target using high-pressure gas and a de-Laval converging-diverging nozzle [1–4]. In this process, a very dense and thick layer can be rapidly deposited without any phase transition or chemical reaction, since the melting of powder caused by a heat source (i.e., arc, plasma, or explosion) is excluded in the process. Instead, the deposition of powder in kinetic spraying is highly dependent on the kinetic energy of supersonic particles. When the particles impact on the target, the extreme kinetic energy contained in the supersonic particles is converted to bonding energy [5–9]. Thus, bonding-related phenomena are quite different from the case of thermal spraying, which involves liquid-state bonding.

Numerous researches were conducted on the fundamental things of

kinetic spraying processes. Early studies reported that the process/ powder conditions affect the deposition-related behavior, closely associated with the material properties of the feedstock powder [10-21]. Several factors are important (e.g., particle/substrate temperature, relative hardness of impact pairs, and oxidation degree), although the most critical issue is whether or not the particle velocity is higher than the critical velocity. Once the required conditions for successful deposition are obtained by using a combination of process/powder parameters and feedstock material properties, some characteristic microstructures are created in the kinetic sprayed deposit (especially, near the particle/particle and particle/substrate interface regions) [22-35]. It has been reported that grains near the bonding interface become refined by grain refinement and static/dynamic restoration [23–31], and that nanoscale phase formation and transformation can be highly localized near the bonding interface [32,33]. Several studies have also investigated distinctive nanoscale mechanical properties of kinetic

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sprayed coating layers [36–39]. These studies have shown that the mechanical properties of the interface region were outstanding, compared to the bulk material, owing to the characteristic nanoscale microstructures in the region.

However, several issues in the kinetic spraying of metals remain unaddressed. In particular, previous studies were somewhat biased to only a few kinds of metals, such as Cu, Al, Ni, and Ti. For the case of copper, various fundamental issues of kinetic spraying were elucidated, from the characteristic deposition behavior [12,17,18,20,21] to the unique microstructural evolution [22,23,28,29,35] and their mechanical properties [39]. Al, which has a low melting point (660 °C) and high ductility, was also frequently studied in kinetic spraying. Particularly, the microstructural evolution of kinetic sprayed Al deposits was intensively investigated by Borchers et al. [28], Kang et al. [24,34], Wang et al. [30], and Balani et al. [31]. It was revealed that grains near the bonding interface become refined by restoration phenomena, such as static recovery/recrystallization [24,28]. During the process, microstructural changes were determined in accordance with the response of Al deposits to the given impact conditions, reflecting the material properties of Al (i.e., dislocation movement and stacking fault energy) [28]. For the case of Ni, Bae et al. [13,14] elucidate the correlation of impact conditions with bonding, nanocrystal formation, and mechanical properties for kinetic sprayed Ni coating layer. It was very impressive that nanotwins could be induced at the bonding interface region of the kinetic sprayed Ni coating layer, despite the high stacking fault energy of Ni compared to that of other metals [40]. In addition, Zou et al. [26,29] reported that a necklace-like structure was created in deposited Ni particles, in which refined grains were located along the particle/particle bonding interface and the grains of the inner particle region remained in a coarse state. Ti was also dealt with in several studies, although it belongs to the hard metals that are difficult to deposit via kinetic spraying. The overall fundamentals of Ti kinetic spraying were examined, from the impact phenomena and bonding features [41] to the microstructural characteristics [25,42], as well as its chemical and mechanical properties [43,44].

In contrast, only a few studies investigated the kinetic spraying of Ta, which is a heavy refractory metal (melting point: 2996°C) possessing BCC crystal structure. Previous studies have shown that kinetic sprayed Ta has a dense and impermeable microstructure and resultant superior corrosion resistance compared to those of thermally sprayed Ta [45-47], assisted by high density, low heat capacity, and good ductility of kinetic sprayed Ta. It has also been reported that moderately annealed kinetic sprayed Ta can be successfully applied to gun barrel liners via explosive cladding of consolidated Ta [48]. Although some microstructural [45] and mechanical [38] characterizations have been performed, more systematic studies in accordance with particle impact conditions are currently lacking in the literature. Owing to the weakening of thermal softening effect at high temperature for BCC metals related to the Peierls-Nabarro (short range) barriers [49], it is quite challenging to reveal the deposition characteristics and related microstructural evolution with mechanical properties. With regard to this, a few studies have investigated the mechanical behaviors of Ta under high-strain and/or high-strain-rate conditions [49-51]. According to Nemat-Nasser et al. [49], the adiabatic flow stress in a Ta Hopkinson bar was controlled by long-range plastic-strain-dependent barriers and short-range thermally activated Peierls-Nabarro barriers, when the high-strain-rate modified Hopkinson technique was applied. In addition, the shear localization behavior, an important factor in kinetic spray processes, of Ta was studied by Chen et al. [50,51]. They suggested that the degree of shear localization is affected by the given strain rate, stress, and temperature, in which the shear localization was enhanced along with the increase in the strain-rate, temperature, and applied shear stress. Accordingly, Ta is expected to show characteristic features in the kinetic spray process.

In this study, the deposition features, related microstructural evolution and mechanical properties of kinetic sprayed Ta under various

 Table 1

 Chemical composition (wt%) of the pure tantalum powder.

N	С	Н	Mg	0	Ta
0.044	0.019	0.075	0.048	0.396	Bal.

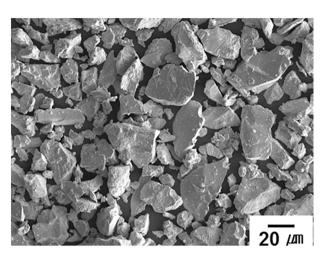


Fig. 1. Scanning electron micrograph of Ta powder.

process conditions were investigated by experiments coupled with finite element modeling (FEM). We believe that the coupled studies discussed herein will provide a fundamental understanding of Ta kinetic spraying, in terms of the processing-structure-property relationships.

#### 2. Methods

# 2.1. Experimental Methods

### 2.1.1. Materials

A commercially available pure Ta powder (Amperit 151.065, H.C. Starck) [45] with particsssle size range  $-30+10\,\mu m$  was used as a feedstock, as shown in Table 1 and Fig. 1.

### 2.1.2. Kinetic Spraying

A kinetic spraying system (KINETICS 3000, CGT, Germany) equipped with a powder preheating system [52] was utilized. The particle temperature can reach the preheating temperature regardless of the particle size (typically 25–75  $\mu m$ ) and the process gas temperature, owing to sufficient heat-up of the particles passing through the elongated coil tube (~6.92 m in length) in the preheater (under  $N_2$  shroud gas environment), which is attached separately to the kinetic spraying system. A de-Laval-type converging-diverging MOC nozzle with a round exit was applied. A number of spray coating experiments were performed onto grit-blasted mild steel substrates (sandblasted using 350  $\mu m$  alumina grits prior to spraying). A 30 mm stand-off distance from the nozzle exit to the substrate was employed. The spray coating experiments were carried out with a powder feed rate of 23.7 g min  $^{-1}$  and gun traveling speed of 80 mm s  $^{-1}$ , under different process conditions, as shown in Table 2.

## 2.1.3. Microstructural Characterization

To examine the porosity and microstructure of the coatings, cross-sections were taken perpendicular to the coating/substrate interface, and subsequently polished to a  $0.3\,\mu m$  alumina finish. Then, all specimens were ultrasonically cleaned and degreased in methyl alcohol for  $60\,s$ . Some specimens for Ta were etched using a solution of  $125\,m L$   $H_2SO_4$ ,  $50\,m L$  HNO $_3$ , and  $50\,m L$  HF for  $15\,s$ . The cross-sectional microstructures and surface morphologies of the coatings were observed

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